REGION 5 RAC2

REMEDIAL ACTION CONTRACT FOR

Remedial, Enforcement Oversight, and
Non-Time Critical Removal Activities at Sites of Release or
Threatened Release of Hazardous Substances in Region 5

FINAL
QUALITY ASSURANCE PROJECT PLAN
Old American Zinc Plant Superfund Site,
Fairmont City
St. Clair County, Illinois
Remedial Design
WA No. 224-RDRD-B5A1/Contract No. EP-S5-06-01

June 2017

PREPARED FOR

U.S. Environmental Protection Agency

PREPARED BY

ch2m.

Ecology and Environment, Inc.
Environmental Design International, Inc.
Teska Associates, Inc.

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Prepared for

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ch2m

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Acronyms and Abbreviations

A2LA	American Association of Laboratory Accreditation
ASL	Applied Sciences Laboratory
bgs	below ground surface
CH2M	CH2M HILL, Inc.
CCV	continuing calibration verification
CL	control limit
CLP	Contract Laboratory Program
COC	contaminants of concern
CRL	Chicago Regional Laboratory
DQO	data quality objective
EDD	electronic data deliverable
EPA	U.S. Environmental Protection Agency
FA	Facility Area
FOP	field operating procedure
FS	feasibility study
GIS	geographic information system
IC	institutional control
ICAL	initial calibration
IDW	investigation-derived waste
IVBA	In Vitro Bioaccessibility Assay for Lead
LCS	laboratory control sample
LDR	Linear Dynamic Range
MDL	method detection limit
ME	marginal exceedance
mg/kg	milligrams per kilogram
MS	matrix spike
MSD	matrix spike duplicate
MPC	measurement performance criteria
NA	not applicable
OAZ	Old American Zinc Plant Superfund Site
PAL	project action limits
PDI	predesign investigation
PM	program manager
ppm	parts per million
PRG	Preliminary Remediation Goal
PRP	Potentially responsible party
QA	quality assurance
QAO	quality assurance officer
QAM	quality assurance manager
QAPP	quality assurance project plan
QC	quality control
QL	quantitation limit
RA	remedial action
RCRA	Resource Conservation and Recovery Act
RI	remedial investigation
RD	remedial design

RPD	relative percent difference
ROD	Record of Decision
RSD	relative standard deviation
site	Old American Zinc Plant Superfund Site
SM	site manager
SOP	standard operating procedure
SSC	site safety coordinator
TBD	to be determined
TCRA	Time-Critical Removal Action
UFP	Uniform Federal Policy
WAM	Work Assignment Manager
XRF	X-ray fluorescence
XTRA	XTRA Intermodal, Inc.

Worksheets #1 and #2—Title and Approval Page

Site Name/Project Name: Old American Zinc (OAZ) Plant Superfund Site **Revision Number:** 0

Site Location: Fairmont City, St. Clair County, Illinois **Site Number/Code:** IL0000034355

Document Title: *Quality Assurance Project Plan, Old American Zinc Plant Superfund Site—Remedial Design*

Lead Organization: U.S. Environmental Protection Agency (EPA)

Contractor Name: CH2M HILL, Inc. (CH2M) **Contractor Number:** EP-S5-06-01

Contract Title: Remedial Design (RD) **Work Assignment Number:** 224-RDRD-B5A1

Preparer's Name and Organizational Affiliation: Shane Lowe/CH2M

Preparer's Address, Telephone Number, and E-mail Address: 300 Hunter Avenue, Suite 305, St. Louis, Missouri, 63124; 314-335-3024; Shane.Lowe@ch2m.com

Preparation Date (Day/Month/Year): April 7, 2017

1. Identify regulatory program:

   The Comprehensive Environmental Response, Compensation, and Liability Act of 1980, commonly known as Superfund

2. Identify approval entity: EPA Region 5
3. The quality assurance project plan (QAPP) is (select one): ☐Generic ☒Project-Specific
4. List dates of scoping sessions that were held: January 19, 2017
5. List dates and titles of QAPP documents written for previous site work, if applicable:

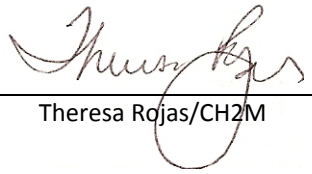
   Not applicable.

6. List organizational partners (stakeholders) and connection with lead organization:

   EPA Region 5 (Lead Organization)
   Illinois EPA (State lead)
   CH2M (Contractor)

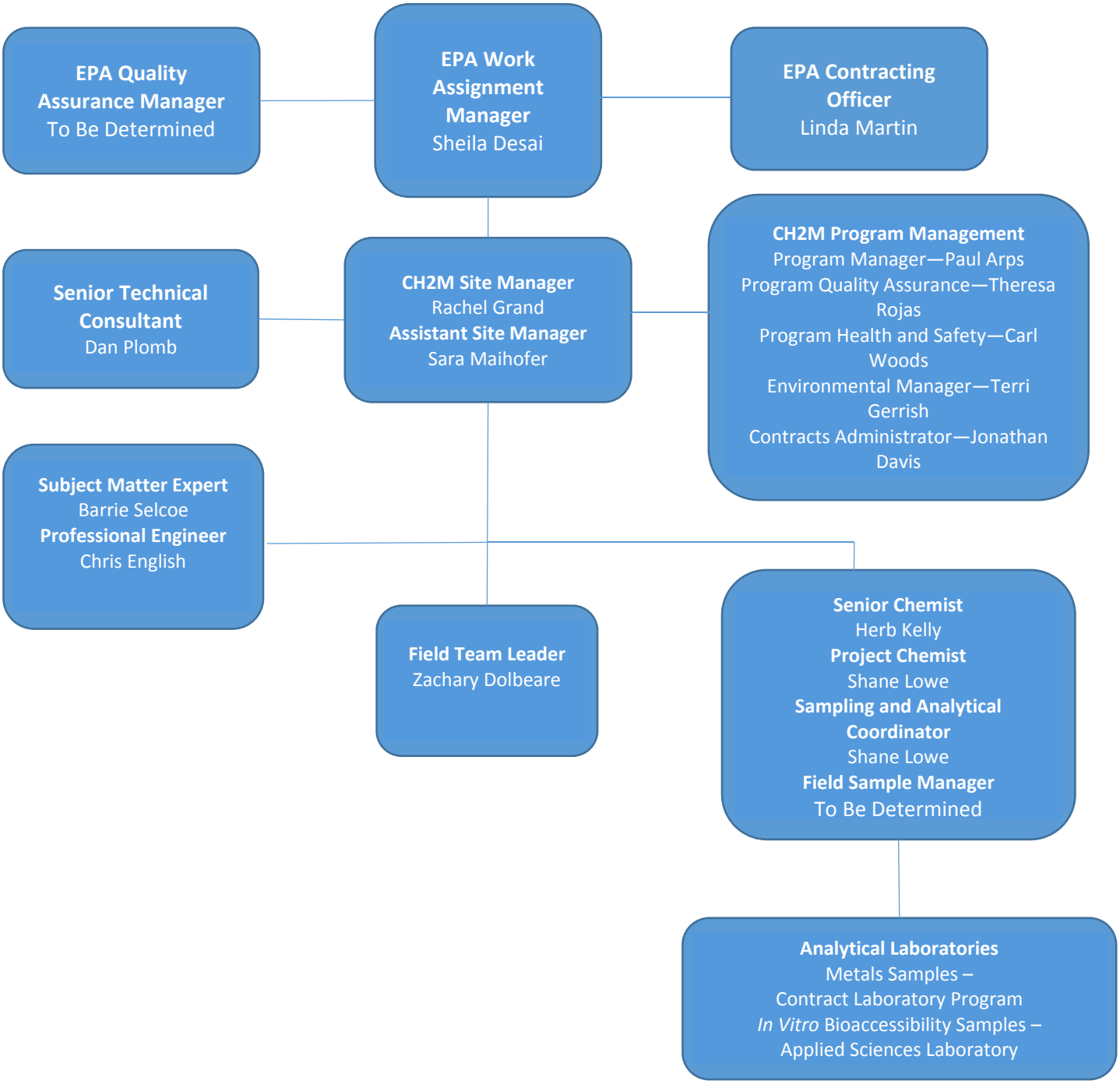
7. List data users:

   EPA, CH2M

Investigative Organization's Site Manager	[Date]	Rachel Grand/CH2M
Investigative Organization's Site Program Quality Assurance (QA) Manager	5/17/17	 Theresa Rojas/CH2M
Investigative Organization's Project Chemist	[Date]	Shane Lowe/CH2M
Lead Organization's Site Project Manager	[Date]	Sheila Desai/EPA
EPA Region 5 QAPP Reviewer	[Date]	Alida Roberman/EPA
Laboratory Director	[Date]	Laboratory Director
Laboratory QA Manager	[Date]	Laboratory QA Manager

Worksheets #3 and #5—Project Organization and QAPP Distribution-1

OAZ Project Organization Chart



Worksheets #3 and #5—Project Organization and QAPP Distribution

OAZ Uniform Federal Policy (UFP)-QAPP Recipients

Note: Not all project staff will receive copies of the UFP-QAPP, but it will be available to them as-needed.

QAPP Recipients	Title	Organization	Telephone Number	E-mail Address
Sheila Desai	Region 5 Work Assignment Manager (WAM)	EPA	312-353-4150	desai.sheila@epa.gov
Alida Roberman	QAPP Reviewer	EPA	312-886-7185	roberman.alida@epa.gov
Paul Arps	Remedial Action Contract Program Manager (PM)	CH2M	414-847-0259	Paul.Arps@ch2m.com
Theresa Rojas	Remedial Action Contract Program QA Manager (QAM)	CH2M	678-530-4297	Theresa.Rojas@ch2m.com
Rachel Grand	Site Manager (SM)	CH2M	314-335-3018	Rachel.Grand@ch2m.com
Sara Maihofer	Assistant SM	CH2M	414-847-0243	Sara.Maihofer@ch2m.com
Kimberly Amley	Deputy Program QAM	CH2M	248-412-7532	Kimberly.Amley@ch2m.com
Herb Kelly	Senior Chemist	CH2M	352-384-7100	Herb.Kelly@ch2m.com
Dan Plomb	Senior Technical Consultant	CH2M	414-847-0222	Dan.Plomb@ch2m.com
Barrie Selcoe	Subject Matter Expert	CH2M	281-721-8527	Barrie.Selcoe@ch2m.com
Chris English	Professional Engineer	CH2M	314-335-3012	Chris.English@ch2m.com
Carl Woods	Health and Safety Manager	CH2M	513-889-5771	Carl.Woods@ch2m.com
Shane Lowe	Project Chemist/Sampling and Analytical Coordinator	CH2M	314-335-3024	Shane.Lowe@ch2m.com
TBD	Project Manager	CLP Laboratory TBD	--	--
TBD	Project Manager	Laboratory TBD	--	--

TBD = To Be Determined

CLP = Contract Laboratory Program

Worksheets #4, #7, and #8—Personnel Qualifications and Signoff Sheet

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Sheila Desai	Region 5 WAM	EPA	Overall responsibility for all phases of work, review, and approval.	
Alida Roberman	Region 5 QAPP Reviewer	EPA	QAPP review and approval.	
Paul Arps	Remedial Action Contract PM	CH2M	Principal point of contact for the EPA contracting officer and project officer. The PM provides leadership and direction to the program and project staff. The PM directs the creation and/or implementation of program policies and procedures to satisfy company policies and client contract specifications, including the preparation of work and quality plans. Selects the program QA manager with endorsement by the business group federal sector quality manager. Reviews budget, schedule, and performance reports. Reviews corrective actions and lessons learned to assess the effectiveness of resolutions. Allocates resources for quality management:	B.S., Chemistry, 15 years of experience
Theresa Rojas	Remedial Action Contract Program QAM	CH2M	Accountable for the overall QA of the program. Approves and charters project quality manager. Evaluates project quality requirements and supports implementation to meet quality requirements. Resolves disputes concerning quality through discussion and negotiation. Reviews and approves the program quality management plan, project QAPPs, program-level quality work instructions and procedures, and project-level documentation based on the project risk and technical management level assignment. The QAM evaluates work assignments and project quality requirements and supports the program technology manager and SMs in project team selection to support the implementation of the project to meet the quality requirements, as needed. The QAM approves project quality managers. The QAM charters project quality managers on quality processes and procedures and supports the development and distribution of quality lessons learned that may arise during the implementation of a project. The QAM prepares and implements the audit plan for the program. The QAM has the authority, as does each team member, to stop work until	B.S., Chemistry, 27 years of experience

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
			corrections are made if there is evidence that work performance will not meet contract specifications, company policy, regulatory requirements, project quality requirements, or if it may result in a hazard to the health or safety of personnel. Disputes concerning the resolution of nonconformance, the type or size of corrective action, or the timeliness of response is forwarded to the QAM. The QAM, project delivery leader, and PM jointly resolve disputes through discussion and negotiation. The QAM has overall dispute resolution authority for RAC2.	
Rachel Grand	SM	CH2M	Responsible for executing all phases of the work assignment and for efficiently applying the full resources of the project team. Responsible for the technical, financial, administrative, and client-related aspects of the project and the project team. Plans the execution of the work assignment and identifies necessary staff. Organizes, directs, and manages personnel and resources. Communicates with the EPA WAM. Responds to and implements corrective actions.	M.S., Geology, 11 years of experience
Dan Plomb	Senior technical consultant	CH2M	Provides the project-specific technology function and is involved throughout the life of the project. Supports the quality and technical accuracy of the work. Monitors the scope, quality, and completeness through consultation and review of project deliverables.	M.S., Geological Engineering, 33 years of experience
Sara Maihofer	Assistant SM	CH2M	Assists the SM with administrative, decision, and approvals. Responsible for the overall technical approach of the project and coordinating deliverable reviews.	B.S., Environmental Engineering, 6 years of experience
Carl Woods	Health and safety manager	CH2M	Responsible for program health and safety, including review and approval of safety plans and subcontractors, monitoring program safety performance, provides incident management and reporting support, and conducts health and safety audits.	M.S., Environmental Safety Management, B.A General Studies, 11 years of experience
Herb Kelly	Senior project chemist	CH2M	Provides senior chemistry expertise.	B.S. Chemistry, 35 years of experience
TBD	Site Safety Coordinator	CH2M	Coordinates, directs, participates in, and reports site activities; ensures adherence to the health and safety plan; communicates issues to SM and field team; contractor oversight. Oversees health and safety for field activities.	

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Shane Lowe	Project chemist	CH2M	Assists in QAPP preparation, coordinates with field team and laboratory, and performs oversight of laboratory and data validation, performs data evaluation.	M.S., Biology, B.S., Wildlife Ecology and Conservation, 18 years of experience
Shane Lowe	Sampling and analytical coordinator	CH2M	Coordinates field and laboratory schedules, and sample management.	M.S., Biology, B.S., Wildlife Ecology and Conservation, 18 years of experience
Kari MacGregor	Database Manager	CH2M	Sets up the project data management system, performs data conversion, quality control (QC) and database maintenance, and prepares data exports (tables, electronic data deliverables [EDDs]).	B.S., Civil Engineering, 19 years of experience
Mark Petershack	Geographic Information System (GIS) Manager	CH2M	Provides GIS support including but not limited to preparation of site-specific geospatial features suitable for EPA Region 5 EDD submission and figure preparation.	B.S., Liberal Arts, 19 years of experience
TBD	Laboratory program manager	Laboratory TBD	Manages sample tracking and maintains communication with the project chemist.	

Special Training Requirements

Project Function	Specialized Training Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
Field Activities	Hazardous waste operations 40-hour training; 8-hour refresher	Registered training organization	Annually	Field staff	Field team staff	CH2M Human Resources Department
Field Activities	Cardiopulmonary resuscitation and first-aid	Registered training organization	Every 3 Years	Site safety coordinator (SSC)	SSC from CH2M	CH2M Human Resources Department
Field Activities	SSC–Hazardous Waste	Registered training organization	Every 3 Years	SSC	SSC from CH2M	CH2M Human Resources Department
Quality	Field quality Manager	CH2M	One time	Field quality manager	Field personnel from CH2M	CH2M Federal Sector Quality Management Department
Site Specific Training	Specified in the health and saftey plan	CH2M	Various	CH2M field personnel working in the field.	CH2M field personnel working in the field.	CH2M Human Resources Department
Health and Safety	Health and safety plan	CH2M	Various	Field personnel and subcontracted personnel working in the field	Field personnel from CH2M and subcontracted personnel	Signoff sheet at the end of the health and safety plan

Signoff Sheet

Organization: EPA

Project Personnel	Title	Telephone Number	Signature	Date
Sheila Desai	Region 5 Work Assignment Manager	312-353-4150		
Alida Roberman	Region 5 QAPP Reviewer	312-886-7185		

Organization: CH2M

Project Personnel	Title	Telephone Number	Signature	Date
Paul Arps	Program Manager	414-847-0259		
Theresa Rojas	QA Manager	678-530-4297		
Rachel Grand	Site Manager	314-335-3018		
Dan Plomb	Senior Technical Consultant	414-847-0222		
Shane Lowe	Project chemist	314-335-3024		

Worksheet #6—Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Communication with CH2M PM	EPA program officer	Linda Martin	312-886-3854	Provides administrative direction to CH2M PM and project team, authorizes changes to plan and can stop work, if needed.
Communication with CH2M contract manager	EPA contracting officer	Michael Dunneback	312-886-7523	Provides administrative direction to CH2M contracts manager.
Communication with EPA program officer and contracting officer	CH2M PM	Paul Arps	414-847-0259	Receives contractual direction from EPA program officer and contracting officer, and notifies EPA of contractual deviations (changes in scope of work, budget, or schedule) by e-mail or letter.
Communication with CH2M SM	EPA WAM	Sheila Desai	312-353-4150	Serves as primary point of contact for EPA, and provides approval of technical direction to CH2M SM.
Point of contact with EPA WAM	CH2M SM	Rachel Grand	314-335-3018	Materials and information about the project will be forwarded to EPA WAM by CH2M SM.
Manage technical project phases	CH2M STC	Dan Plomb	414-847-0222	Notifies SM of RD-related problems by next business day. Serious issues will also be reported to CH2M QAM.
Field staff discussion and inquiry	CH2M field team leader	Zachery Dolbeare	217-416-2540	Serves as a primary point of contact for field team before, during, and after the remediation; communicates back to the SM, project quality manager, and project chemist, as needed. Communication by phone as needed with field staff during field sampling events, followed up with e-mail to document decisions and actions.
Health and safety	CH2M SSC	TBD	--	Responsible for the adherence of team members to the site safety requirements described in the health and safety plan. Will report health and safety incidents and near misses to the SM and health and safety manager.
QAPP changes in the field	CH2M field quality manager	TBD	--	The field quality manager will notify the SM and STC by phone and e-mail of changes to the QAPP made in the field and the reasons within 24 hours. Documentation of deviations from the work plan will be kept in the field logbook; deviations made only with the approval of the contractor SM. The SM will advise the EPA WAM of changes.
Daily field progress reports	CH2M field team leader	Zachery Dolbeare	217-416-2540	E-mail or fax daily field progress reports to CH2M PM.
Field corrective actions	CH2M field quality manager	TBD	--	The need for corrective action for field issues will be determined by the field team leader. The SM will ensure QAPP requirements are met by field staff. The field quality manager will notify the SM of needed field corrective actions. The SM will have 24 hours to respond to the request for field corrective action.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Reporting laboratory data quality issues	CH2M field team leader	Zachery Dolbeare	217-416-2540	QA/QC issues with field samples will be reported to the project chemist immediately.
Analytical corrective actions	Project chemist	Shane Lowe	314-335-3024	The need for corrective action by the analytical laboratory will be determined by the project chemist. The project chemist will ensure QAPP requirements are met by the laboratory. No analytical data can be released until data are reviewed for completeness and conformance to analytical guidelines by the project chemist. The project chemist will review the data as soon as possible upon receipt from the validator.
Release of analytical data	Laboratory project manager	TBD	--	No analytical data can be released to CH2M and EPA until it has been reviewed by the laboratory. No final data can be released to CH2M until validation is completed and the laboratory has approved the release.
QAPP amendments	Region 5 WAM and QAPP reviewer	Sheila Desai, TBD		Major changes to the QAPP must be approved before the changes can be implemented.

Worksheet #9—Project Planning Session Summary

A project scoping session was held on January 19, 2017, for discussion on soil sampling activities and RD for the OAZ Plant Superfund Site (site) located in Fairmont City, Illinois. The teleconference meeting was attended by EPA Region 5 and CH2M representatives.

Site Name:	Old American Zinc Plant Superfund Site
Project Name and Site Location:	Old American Zinc Plant Superfund Site, Fairmont City, Illinois
Projected Date(s) of Field Activities:	Summer 2017
Site Manager:	Rachel Grand

Date of Session: 1/19/2017

Scoping Session Purpose: Discuss site boundary, properties to be included for sampling, schedule of activities, and sampling and design approach for the Old American Zinc Plant Superfund Site located in Fairmont City, Illinois.

Name	Title/Role	Affiliation	E-mail Address
Sheila Desai	WAM	EPA Region 5	desai.sheila@epa.gov
Rachel Grand	EPA Consultant Project Manager	CH2M	Rachel.Grand@ch2m.com

Decisions/Discussion Items

The following items were discussed:

- Sampling Approach:
  - The project boundary was confirmed and properties to be sampled within the boundary included residential, commercial, and vacant properties. During a preliminary review of the parcels within the site boundary, CH2M assumed that up to 300 properties with up to 2 yards each and 31 alleyways will be sampled as part of the 2017 work.
  - It was confirmed that properties that were included in the Time-Critical Removal Action (TCRA) and did not have exceedances will not be resampled.
  - CH2M assumes a 7-person sampling team (6 samplers and 1 sample manager).
  - Investigation-derived waste (IDW) will not be generated. The soil cuttings from shallow soil borings will be placed back in the hole, minimal decontamination water will be created (minimal spraying of equipment), and trash will be disposed of as general refuse.
- Schedule of Activities:
  - June 2017—Conduct a public meeting in Fairmont City, Illinois, with CH2M and EPA present.
  - June 2017—CH2M will complete and mail the access request letters to the property owners.
  - June 2017—CH2M and EPA will go door-to-door in attempt to collect access agreements for property owners that have not responded.
  - July or August 2017—Mobilize for sampling activities.

  - Illinois EPA will review documents concurrently with EPA. CH2M will assume a 45-day review turnaround time for agency review of documents.

- Analytical Approach:
  - The QAPP will be prepared as soon as soon as the work plan is approved to ensure that it is completed and approved prior to the proposed start of fieldwork.
  - The Contract Laboratory Program (CLP) laboratories will be used for the metals analysis. CH2M will work closely with the CLP when scheduling the work and will manage the field sampling schedule based on the capacity of the laboratories. It is estimated that the CLP will submit the data within 60 days of the final sample submittal.
  - CH2M will perform the data validation of the data received from the CLP and upload the data to a site EQuIS database.
  - CH2M will calculate site-specific in vitro bioaccessibility (IVBA) for lead at the residential properties. CH2M will collect IVBA samples from 10 percent of the properties and submit samples to a laboratory. It is assumed that this laboratory is outside of the CLP. Following analysis of the bioaccessibility, a site-specific IVBA value will be calculated by a statistician and a human health risk assessor.
- Design and GIS:
  - The design documents will include drawings of each property with an exceedance, which CH2M will generate.

Worksheet #10—Conceptual Site Model

Site Description

Site Location

The OAZ Superfund Site is located in the Village of Fairmont City in St. Clair County, Illinois. The site includes a 132-acre Facility Area (FA) and surrounding properties, where elevated metal concentrations associated with the facility operation were found in different media. The FA is bordered by several commercial and industrial properties, including Garcia Trucking to the west, CSX Intermodal railroad yard to the south, and General Chemicals to the east. Most the residential properties lie to the west of the FA, with smaller pockets of residential or trailer-park developments to the south, east, and north of the FA. OAZ conducted zinc smelting operations at the site from 1916 to 1967. Slag from the smelting operation was cooled by placing the molten material along the northern and western boundary of the FA. The slag stock piles originally encompassed an area of 15 acres. The site, including the clinker and other smelting residues on the property, was purchased by XTRA Intermodal, Inc. (XTRA), in 1979. XTRA operated a trucking terminal at the site until 2003 that involved the leasing, storage, and maintenance of a diverse fleet of trailers. XTRA ground up and redistributed the slag stockpiles on the FA to build up and level the former plant site to facilitate its trucking operation. At present, redistributed slag on the FA covers an area of 125 acres with thickness ranging from 6 inches to 9 feet (ENTACT 2012).

Site History

Site investigations conducted at the site since 1994 detail the nature and extent of contamination in the FA and surrounding properties. ENTACT completed a remedial investigation (RI) (ENTACT 2009a) and feasibility study (FS) (ENTACT 2012) for the FA in 2012 and identified contaminants in different media that included slag stock piles, ground slag that was used as fill material, and high metal concentrations in shallow groundwater.

The impacted surrounding areas include residential, commercial, and vacant properties, village alleyways, and drainage ways that were contaminated with runoff from the facility. Ground slag was transported to offsite properties by local businesses, residents, and the village for surfacing village alleyways and used as fill material in residential properties (ENTACT 2012). Most the impacted properties are located to the west of the FA, with small pockets of trailer-park and residential developments to the north, south, and east.

EPA, under the provisions of the Comprehensive Environmental Response, Compensation, and Liability Act, conducted a TCRA from 2002 to 2003. A total of 462 offsite properties was sampled during the TCRA, of which 209 properties were found to have lead concentrations above the TCRA Removal Action Limit of 400 parts per million (ppm) for residential properties and 1,000 ppm for commercial properties. Impacted soil was removed from 152 properties, with the remaining properties to be addressed under future remedial action. An additional 25 properties and 8 alleyways were sampled as part of the RI.

Following the completion of the RI/FS in 2012, a Record of Decision (ROD) (EPA 2012) was issued by EPA detailing the selected remedial approach for the site. EPA entered into an Administrative Order on Consent with the potentially responsible party (PRP) in August 2014 to perform the RD work, and ARCADIS was tasked to perform the design. An additional 14 residential properties were sampled as part of the predesign investigation (PDI) (ARCADIS 2016a) and a draft final RD report (consisting of the report, selected drawings, but no technical specifications) (ARCADIS 2016b) was submitted to EPA in April 2016. The entity responsible for the PRP's work filed for Chapter 11 bankruptcy and ceased performing additional work at the site in April 2016. . As a result, EPA took control of the site in order to complete the RD. EPA, under Work Assignment No. 224-RDRD-B5A1, has tasked CH2M to complete the RD activities for the selected remedy, in the FA and offsite properties.

The Problem to be addressed by the Project

The purpose of this soil sampling is to collect additional data to evaluate the nature and extent of the contaminants of concern (COCs) (arsenic, cadmium, zinc, and lead) in offsite residential, vacant, and commercial properties and alleyways associated with the site in order to complete the RD and to determine the site-specific IVBA for lead at residential properties.

The remedy for offsite properties includes the following:

- Soils from residential, commercial/industrial, or vacant properties will be removed up to a maximum depth of 2 feet. If concentrations exceeding the cleanup levels remain at 2 feet depth, a demarcation barrier will be placed at the base of the excavation before backfill and restoration.
- Source material or contaminated soil will be removed from village alleyways to a depth of 2 feet. If the cleanup level exceedance remains below 2 feet, a demarcation barrier will be placed at the base of the excavation before backfill and restoration.
- The excavated material will be placed within a newly constructed consolidation area and capped with a cover system consisting of a 24-inch low-permeability clay barrier, overlain by a 12-inch vegetative soil cover.

Previous soil investigations in the surrounding properties were used to determine if the source material had contaminated the underlying and surrounding area soils. In samples from offsite residential and commercial properties, exceedances in screening criteria were primarily found in areas where slag-like granular material had been placed as fill or surfacing material. Exceedances in alleyways were associated with loose granular slag beneath the compacted gravel layer and concentrations typically decreased rapidly in the native soil below the fill material.

The RI evaluated the airborne deposition of contaminants originating from the FA. The occurrence of contaminants in the offsite properties were not correlated with airborne deposition as no regular pattern of exceedance was found (ENTACT 2009a). Sampling of properties downwind of the FA, to the south and east, did not show elevated concentrations consistent with signification airborne deposition. Exceedances were attributed to redistributed slag on the properties and alleyways. Contaminant migration from the FA occurs from surface water runoff through a series of man-made drainage ditches on the FA to Rose Creek, which ultimately flows into Old Cahokia watershed.

The site is now in the RD phase, which is designed to achieve the remediation goals specified in the ROD. This phase involves detailing the cleanup specifications and design of the selected remediation technology. EPA's selected remedy for the site is Alternative 4A as described in the ROD (EPA 2012). The overall strategy for the site is to contain and cover the low-level threat waste in order to reduce future human health and ecological risk to acceptable levels. The ROD defines "nonresidential" properties to include commercial/industrial properties where institutional controls (ICs) will restrict future residential use and alleyways, manmade ditches/outfalls, and Rose Creek where neither residential nor commercial use are likely to occur.

The selected remedy for the offsite properties involves removal of source material (slag used as fill) and contaminated soil from the identified residential, commercial/industrial, vacant properties, or village alleyways above the applicable residential or commercial/industrial human health cleanup levels. The removed material will be consolidated within a 35-acre consolidation area located in the southwest portion of the FA. The properties will be back filled with clean fill and restored to pre-excavation conditions. The cleanup levels for residential and nonresidential properties are presented in Table 10-1. Cleanup levels were selected from the lower of the calculated noncancer preliminary cleanup goal, and the calculated cancer preliminary cleanup goal for each of the three target risk levels as summarized in the baseline human health risk assessment (ENTACT 2009b) and baseline ecological risk assessment (ENTACT 2008). Residential cleanup levels for arsenic, cadmium, and zinc were developed assuming unrestricted future use. However, a site-specific residential cleanup level for lead was not

evaluated in the RI or the FS. IVBA testing of samples collected during this field investigation will be used develop a risk-based cleanup criteria for lead.

Investigations conducted by the PRP's contractors as part the RI/FS and the TCRA focused on the residential and commercial properties to the west of the FA, of which approximately 80 properties are listed as requiring additional investigation, access denied, or exceeding the preliminary remediation goal (PRGs) in verification samples. EPA has contracted CH2M to collect additional soil data at these previously sampled properties and also to expanded residential soil investigations to properties further north and west of the previously sampled area and further east and south of the FA that were not covered in the previous sampling. Based on discussions with EPA, sampling of up to 300 residential and commercial properties and 31 alleyways will be conducted at the site in order to delineate the extent of the contamination, up to a depth of 24 inches. The field sampling will also include collection of soil samples for the IVBA study for developing a site-specific residential cleanup level for lead. The data obtained from this investigation will be used to complete the RD of the sampled properties and alleyways.

Table 10-1. Final Cleanup Levels for Old American Zinc Plant Site

Contaminants of Concern	Soil/Sediment	
	Residential (mg/kg)	Nonresidential (mg/kg)
Arsenic	32	239
Cadmium	37	809
Lead	400[c]	826
Zinc	6,400	31,852[a]/306,000[b]

[a] Based on Ecological Risk for Sediment

[b] Based on Human Health Risk for Soil/Sediment

[c] EPA may specify a site-specific cleanup level for lead based on the IVBA study; currently using the Remedial Action Limit (RAL) of 400 ppm.
mg/kg = milligrams per kilogram

A Synopsis of Secondary Data or Information from Site Reports

Previous sampling conducted at the offsite residential and commercial/industrial properties include TCRA, RI, and PDI.

- As part of the TCRA, 462 properties were investigated for metals, and removal action was completed on 152 properties. The Remedial Action Limit (RAL) for the TCRA was 400 mg/kg total lead for residential properties and 1,000 mg/kg total lead for commercial/industrial properties. No removal action was performed on vacant properties unless soil sample results exceeded 1,200 mg/kg, and the property posed a significant risk for trespassers and children who may come in contact with the soil.
- Out of 4,400 soil samples collected from 462 TCRA properties, 444 samples from 362 properties were submitted to the laboratory for arsenic, cadmium, lead, and zinc analysis, and the remaining 100 properties were screened using X-ray fluorescence (XRF).
- As part of the RI, 25 properties and 8 alleyways were sampled. Eleven of the 25 properties were access-denied properties from the TCRA investigations. A total of 517 composite residential, vacant, and commercial soil samples were screened using XRF and approximately 10 percent of the samples were submitted to laboratory for Resource Conservation and Recovery Act (RCRA) Eight metals analysis (arsenic, bromine, cadmium, chromium, lead, selenium, silver, and mercury) and zinc. Seventeen of the 25 properties contained soil concentrations exceeding the PRGs. A total of 176 composite samples were collected from the eight alleyways at depths of 0 to 2 feet below ground surface (bgs) and screened using XRF. Ten percent of the composite samples were submitted for laboratory analysis of RCRA Eight metals analysis and zinc.

- In the PDI, 132 samples were collected from 14 properties, and all samples were submitted to the laboratory for arsenic, cadmium, lead, and zinc analysis.
- The RD completed by ARCADIS (ARCADIS 2016b) identifies 77 properties, including 12 commercial properties, as requiring remedial action.
- The master database provided by the PRPs lists the following:
  - 74 properties are listed as to be remediated including 2 alleyways, 11 commercial, and 61 residential properties
  - 4 properties are listed as to be remediated based on PDI results
  - 5 properties are listed as access denied
  - 59 properties are listed as more data needed, including 6 alleyways and 53 residential properties
  - 12 properties are listed as exceeding PRGs in post-verification samples

In the additional residential and alleyway soil sampling to be conducted by CH2M, an estimated 300 properties and 31 alleyways will be sampled. These properties will include residential properties that were listed as requiring more data, access-denied, or exceeding PRGs in verification samples.

Sampling Rationale

The COCs (arsenic, cadmium, zinc, and lead) were identified in the previous site investigations (RI report [ENTACT 2009a]). Soil samples will be collected to evaluate the nature and extent of the contamination in properties that require additional data and properties not previously sampled. The data will be used to identify excavation depths for those properties with cleanup level exceedances. A site-specific residential cleanup level for lead has not be established for the site. The results of the IVBA sampling to be conducted as part of this data collection effort, will be used to develop a site-specific risk-based cleanup criterion for lead in residential properties. The activities involved in collection of the additional data for the RD will consist of the following:

- Coordinate access with property owners.
- Subcontract and coordinate with an analytical laboratory and third party utility locator.
- Mobilize to the site.
- Locate and mark underground utilities.
- Collect surface and subsurface soil samples from residential, vacant, and commercial properties and alleyways with access to determine if properties need remediation and/or identify the vertical extent of remediation.
- Submit soil samples to a CLP offsite laboratory for total arsenic, cadmium, zinc, and lead analysis.
- Collect surface soil samples from up to 30 yard sections for the IVBA study.
- Submit soil samples to a project-approved laboratory for IVBA testing.
- Prepare interim deliverables and data evaluation report.
- Demobilize.

Project Decision Conditions (“If…, then…” statements)

Refer to Table 11-1, Data Quality Objectives.

Worksheet #11—Project/Data Quality Objectives

Table 11-1. Data Quality Objectives (DQOs)

DQO #	Step 1: Statement of Problem	Step 2: Identify Goals of the Study	Step 3: Identify Information Inputs	Step 4: Define Boundary Studies	Step 5: Develop Analytical Approach	Step 6: Specify performance or acceptance criteria	Step 7: Develop plan for obtaining data
1	*Problem:* Site-related COCs (arsenic, cadmium, zinc, and lead) have been previously identified in surface and shallow soil of residential, commercial, and vacant properties and alleyways. Horizontal and vertical extents of soil impacted with arsenic, cadmium, zinc, and lead needs to be determined for the remedial action. *General intended use of collected data:* The data in this study will be used to evaluate the current concentrations and extents of arsenic, cadmium, zinc, and lead in soil at up to 300 properties and 31 alleyways that were not sampled previously, were only partially sampled or were previously screened using sample screening methodology.	*Principal Study Question:* Determine horizontal and vertical extent of surface and subsurface soil contamination to develop an RD. *Range of Possible Outcomes:* • Data are sufficient to define the extent of elevated COC concentrations at each property sampled. • Data are inconsistent with the conceptual site model and additional characterization is required.	*Information Needed to Resolve the Decision Statement:* • Collect surface and shallow subsurface soil samples from required areas at up to 300 properties and 31 alleyways. *Source of information:* Data were obtained from the *Draft Final Design Report* (ARCADIS 2016b) *Parameters/characteristics to be measured:* Arsenic, cadmium, zinc, and lead *Sampling and analysis method:* Based on guidance from the EPA *Superfund Lead-Contaminated Residential Sites Handbook,* the number of composite sampling locations per residential, commercial and vacant property will be determined by the surface area of each property (up to three 5-point composites will be collected from properties less than 5,000 square feet, and 4 or more 5-point composites will be collected for properties greater than 5,000 square feet). Soil sampling will consist of 5-point composite sampling per yard area. The 31 alleyways will be sampled using a 5-point composite per 2,500-square-foot grid.	*Spatial:* Previous RI did not address all potentially COC-impacted properties, which resulted in up to 300 residential, commercial, and vacant properties and 31 alleyways that require further investigation. *Temporal:* Sampling will be performed to assess the current concentrations and spatial extents of COCs.	Soil samples will be analyzed for arsenic, cadmium, zinc, and lead. Refer to Worksheet #17.	The data should identify the concentrations and locations of COCs enough to support the development of a data evaluation report and property drawings for the design where remediation is required. Soil remedial goal is 32 mg/kg (residential) and 239 mg/kg (nonresidential) for arsenic, 37 mg/kg (residential) and 809 mg/kg (nonresidential) for cadmium, 400 mg/kg (residential) and 826 mg/kg (nonresidential) for lead, and 6,400 mg/kg (residential) and 31,852/306,600 (ecological nonresidential/human-health nonresidential) for zinc. Refer to Worksheets #24, #28, and #36 for acceptance and performance criteria. Laboratory data are considered usable if data validation criteria are met (refer to Worksheet #37 for data usability criteria).	Collect surface and shallow subsurface soil from up to 300 properties using a 5-point composite sampling approach and 31 alleyways using a 5-point composite sampling approach for each 2,500 square feet. Soil samples will be analyzed for arsenic, cadmium, zinc, and lead. Refer to Worksheet #17 for details on the sample collection design and rationale.
2	*Problem:* Evaluate the need for a site-specific residential cleanup level for lead. *General intended use of collected data:* The data in this study will be used to evaluate the IVBA of the lead in the site soil and, which could assist the EPA in developing a site-specific residential cleanup level for lead.	*Principal Study Question:* Conduct a site-specific lead IVBA study. *Range of Possible Outcomes:* • Data are sufficient to define the IVBA of the lead onsite. • Data are inconsistent with the conceptual site model and additional characterization is required.	*Information Needed to Resolve the Decision Statement:* • Collect surface soil samples from one yard area each at up to 30 properties. *Source of information: Guidance for Sample Collection for In Vitro Bioaccessibility Assay for Lead (Pb) in Soil* (USEPA 2015). *Parameters/characteristics to be measured:* fraction of lead solubilized from a soil sample *Sampling and analysis method:* Soil sampling will consist of collecting one 30-point composite sampling at one yard area per property at up to 30 yards using a hand trowel or equivalent.	*Spatial:* Sampling will be performed from one yard area at up to 30 residential properties. *Temporal:* Sampling will be performed to determine the fraction of lead in the soil matrix absorbed by an organism by a specific exposure pathway.	Soil samples will be analyzed to measure the fraction of lead solubilized from a soil sampling under simulated gastrointestinal conditions. Refer to Worksheet #17.	The data in this study will be used to evaluate the IVBA of the lead in onsite soil and assist the EPA in developing a site-specific residential cleanup level for lead. Refer to Worksheets #24, #28, and #36 for acceptance and performance criteria. Laboratory data are considered usable if data validation criteria are met (refer to Worksheet #37 for data usability criteria).	Collect surface soil from up to 30 properties using a 30-point composite sampling approach. Soil samples will be analyzed to measure the fraction of lead solubilized from a soil sampling under simulated gastrointestinal conditions. Refer to Worksheet #17 for details on the sample collection design and rationale.

11.1 Who will use the data?

The analytical data will be used by CH2M and EPA.

11.2 What will the data be used for?

Soil sample collection and other associated tasks will be conducted to fill data gaps necessary to complete the RD, assuming 50 percent of the properties sampled will exceed the cleanup goals, the design will include up to 150 properties and 15 alleyways and calculate the site-specific cleanup level for lead using the IVBA study results. The data collected as part of the action will be used to meet the DQOs.

11.3 What types of data are needed?

Task	Sampling Activity/Objective	Sampling Frequency/ Duration	Matrix	Parameters
Soil	Arsenic, cadmium, zinc and lead using 5-point composite sampling per yard and per 2,500 square feet in alleyways	Once	Soil	Arsenic, cadmium, zinc, and lead
Soil	IVBA 30-point composite sample per yard area	Once	Soil	Fraction of lead solubilized from a soil sample and total lead; samples sieved in the laboratory with a 150-micron sieve

11.4 How "good" do the data need to be in order to support the environmental decision?

The data should meet the project action levels as specified in QAPP Worksheet #15 and the QC requirements that are explained in QAPP Worksheet #37.

11.5 How many data are needed? (Number of samples for each analytical group, matrix, and concentration)

Worksheet #17 (Sampling Design and Rationale) describes the field investigation activities. Worksheet #18 (Sampling Locations and Methods) summarizes the number of samples and the analytical parameters.

11.6 Where, when, and how should the data be collected/generated?

Detailed information on where, when, and how the data will be collected is provided in Worksheets #17 and #18.

11.7 Who will collect and generate the data?

CH2M will collect environmental samples. The subcontracted analytical laboratory and CLP laboratory will generate the data results for all samples. The subcontracted analytical laboratory SOPs will be provided as Appendix B upon laboratory procurement.

11.8 How will these data be reported?

The data will be reported in accordance with the procedures outlined in Worksheets #34, #35, #36. Observations of soil and site activities will be recorded in project-specific logbooks.

11.9 How will these data be archived?

The final evidence file will be the central repository for all documents that constitute evidence relevant to sampling and analysis activities. CH2M will be the custodian of the evidence file and will maintain the contents of the evidence files for the project, including relevant records, reports, logs, field notebooks, sketches, pictures, contractor reports, and data reviews in a secured area with limited access. CH2M will keep all records for 10 years after contract completion. As necessary, records may be transferred to an offsite records storage facility.
The records-storage facility must provide secure, controlled-access records storage.

Worksheet #12—Measurement Performance Criteria

Measurement performance criteria (MPC) were established for soil analytical parameters for the project. The criteria relate to data quality indicators consisting of precision, accuracy, representativeness, comparability, completeness, and sensitivity, commonly referred to as PARCCS parameters.

In order to increase the document efficiency and reduce redundancy, refer to the following worksheets for the required information in Worksheet #12:

- Worksheet #15 (Reference Limits and Evaluation) and Worksheet #37 (Data Usability Assessment) for laboratory and field data quality indicators consisting of precision and accuracy
- Worksheet #24 (Analytical Instrument Calibration) and Worksheet #28 (Analytical Quality Control and Corrective Action) for the requirements of laboratory QA/QC activities for soil analytical methods
- Worksheet # 35 (Data Verification Procedures) and Worksheet #36 (Data Validation Procedures) for data review and validation process
- Worksheet #37 (Data Usability Assessment) for PARCCS parameters

The quality of the data to be collected for this project will be verified using appropriate MPC established for both sampling procedures and analytical methods. The MPC follow those defined in the referenced EPA method or laboratory standard operating procedure (SOP). The quality of the sampling procedures and laboratory results will be evaluated for compliance with project DQOs through a review of overall PARCCs, in accordance with procedures described in Worksheet #37 (Data Usability Assessment). The results will be summarized in an overall data quality report, which will be included as an appendix to the data evaluation report.

Worksheet #13—Secondary Data Uses and Limitations

Secondary data refer to historical data previously collected. The source(s) of the data, date of collection, planned uses, and limitations of the secondary data for each site are summarized in the following table.

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates)	How Data Will Be Used	Limitations on Data Use
• Arsenic, cadmium, lead, and zinc results collected from residential, commercial and vacant properties and alleyways.	ARCADIS. *Draft Final Design Report*. 2016.	A total of 462 residential, commercial, and vacant properties located in Fairmont City and the adjacent community of Washington Park were sampled for lead, arsenic, cadmium, and zinc as part of a 2002-2003 TCRA conducted at the site. Thirty-nine residential, commercial, and vacant properties and eight alleyways were sampled as part of the RI (2009) and PDI (June 2015).	Analytical results will be used in identifying the yard areas requiring remediation.	Additional sampling and analysis will be required to determine the remediation depth in the yard and which yard areas.
• Arsenic, cadmium, lead, and zinc results collected from residential, commercial, and vacant properties and alleyways.	ENTACT. Final Remedial Investigation Report. 2009. (Including data from the Final Removal Action Report [ENTACT 2003]).	The primary source of metals concentrations at the site was determined to be localized stockpiles of vitrified slag material and ground, granular slag material re-distributed across the FA. Composite samples from residential, commercial, and vacant properties and alleyways were collected and screened with XRF to delineate arsenic, cadmium, zinc, and lead concentrations in the soil at residential properties where access had been obtained. Thirty-nine residential, commercial, and vacant properties and eight alleyways were sampled. Ninety-two sediment samples were collected from ephemeral drainage ditches and Rose Creek, and 34 surface water samples were collected from ephemeral ditches, Rose Creek, Schoenberger Creek, and the Old Cahokia Watershed and analyzed for total and dissolved RCRA eight metals. Five samples of slag were collected for toxicity characteristic leaching procedure testing.	Analytical results will be used in identifying the yard areas requiring remediation. Analytical data will also be used to select properties for the IVBA study.	Additional sampling and analysis will be required to determine the remediation depth in the yard and which yard areas.

Worksheets #14 and #16—Project Tasks and Schedule

Combined Worksheets #14 and #16 provide an overview of project tasks as the outcome of project scoping activities and includes a project schedule (Worksheet #15). The following project tasks are discussed:

- Field/sampling tasks
- Laboratory analysis
- Data management
- Data review and usability
- Reporting

Fieldwork/Sampling Tasks

Applicable field operating procedures (FOPs) for the project tasks outlined in this worksheet are listed on Worksheet #21 and provided in Appendix A.

- Mobilization activities associated with preparation for soil sampling, IVBA study, alleyway sampling, and sketching activities.
- Third-party private utility locate at residential, commercial, and vacant properties and alleyways with proposed sampling.
- Advancement of soil borings with a hand auger to a maximum depth of 2 feet bgs. Five soil borings will be located to satisfy a 5-point composite per yard area (front, back and side) at each property. Up to 1 composite soil sample per 6-inch intervals from up to 4 depth intervals will be collected per yard area at each property. For properties under 5,000 square feet, up to 12 samples per property will be collected and for properties greater than 5,000 square feet up to 16 samples per property will be collected.
- Advancement of soil borings with a direct-push technology rig to a maximum depth of 2 feet bgs. Soil borings will be located to satisfy a 5-point composite per 2,500 square foot area in each alleyway. A maximum of 16 composite soil samples are estimated per alleyway, assuming 4 composite samples with one from each of 4 depth intervals will be collected per alleyway.
- Collecting surface soil samples to a maximum depth of 1 inch bgs. Surface soil samples will be located to satisfy a 30-point composite for one yard area (front yard or back yard) at up to 30 yards.

Analysis Tasks

Laboratory analyses are described in Worksheet #17 (Sampling Design and Rationale) and are summarized in the following paragraphs. Samples will be analyzed as described in Worksheet #15 (Reference Limits and Evaluation). Soil samples will be analyzed for select metals by Method ISM02.4. In addition, select soil sample locations will be analyzed for lead IVBA based on Method 1340. The metals samples will be analyzed by a CLP laboratory. Applied Sciences Laboratory (ASL) in Corvallis, Oregon, will analyze the bioaccessability samples. The laboratory analyses will be performed in accordance with the analytical methods, this UFP-QAPP, and the laboratory SOPs as defined in Worksheet #23 (Analytical SOPs).

Quality Control Tasks

Implement SOPs. For items related to QC, see Worksheets #11, #12, #15, #22, #24, #25, #27, and #28.

Secondary Data

See QAPP Worksheet #13.

Data Management Tasks

The data will be tracked, stored, handled, and managed. Field activities will be recorded in project logbooks and on applicable field forms. Site maps will be maintained and sample locations will be updated on the maps as necessary. Field and analytical data will be consolidated and maintained within an electronic database management system. The database management system will be used to perform sample tracking, storage of electronic data, validation of data, querying data for analysis, and preparation of final data tables.

The following are the team members and their responsibilities for the data management process:

- **Project Chemist—**Responsible for reviewing the chain-of-custody forms and establishing the sample tracking system weekly. Oversees proper use of EPA's sample management system (Scribe) and accuracy of the information entered. Reviews laboratory data for accuracy and quality and compares electronic outputs for accuracy to laboratory electronic copies. Conducts tracking of samples, forwards tracking information and received data to the database manager, and identifies the data inputs (for example, sample numbers) to use in generating tables and plots. Assists with logistical support of the field team.
- **Database Manager—**Responsible for setting up the data management system in consultation with the project chemist at the beginning of the data evaluation task. Also oversees the data management process, including data conversion/manual entry into the data management system, QC of the entered data, and preparation of the required tables and plots of the data. Coordinates upload of EDMAN deliverable. Coordinates with the person responsible for reviewing the entered data for QC purposes. Forwards deliverables to the SM.
- **GIS Manager—**Responsible for coordinating with the SM to set up the geodatabase prior to sampling. Maintains spatial layers and overall geodatabase integrity and accuracy. Provides GIS-related outputs for reports and data management.

Sample Tracking

The project chemist is responsible for tracking samples in the sample tracking database to ensure that the analytical results for all samples sent for analysis are received. Copies of chains of custody from the field team are used to enter in sample IDs, collect date, and analyses. Upon receipt of a sample receipt notice from the laboratory, the date received by the laboratory, and a date the electronic copy is due will be entered. Likewise, upon receipt of the electronic copy and EDD, the date they were received will also be entered. The EDDs will be uploaded when received from the laboratory and will be tracked in the sample tracking table. Validation qualifiers will be added to the database and results qualified accordingly.

Data Types

The data will be added to the project database as they become available. The data will include Scribe field data and new data collected in the laboratory. The Scribe field data will be exported and loaded into the Sample Tracking database by the project chemist. The EPA's contractor will validate the metals data and the IVBA data will be validated by CH2M. The data source will be noted in the database.

Data Tracking and Management

Every data set received from analytical laboratories will be tracked individually. Analytical laboratory reports of chemical analysis results will be tracked in a consistent fashion. Every data set will be assigned a unique identifier. The date of receipt, status of data validation, and status of database entry for each data set will all be tracked and recorded in the project database.

Hard/Electronic Copy

Measurements made during field data collection activities will be recorded in field logbooks and field forms. Field data will be reduced and summarized, tabulated, and stored along with the field logbooks and sample processing logs. Scans of field data will be stored electronically in the project file.

The raw analytical laboratory data are stored electronically.

Data Input Procedures

Sampling information, analytical results, applicable QA/QC data, data validation qualifiers, and other field-related information will be entered into the project database for storage and retrieval during data evaluation and report development. The analytical data will be loaded into the database using EDD files received from the analytical laboratory. Validation qualifiers will be entered manually. Other available field-related data collected will be manually entered onto standard EDD templates for loading into the database. Historical data, either in hard copy or electronic form, will be manually entered or formatted to standard EDD templates for database loading.

Computer Database

The technical data, field observations, laboratory analytical results, and analytical data validation will be managed by CH2M in their data management system using EQuIS 6, a third-party database system by EarthSoft, Inc. The EQuIS 6 data management system is used in EPA Region 5 to store and analyze project data submissions. CH2M will manage data in a manner consistent with EPA Region 5 guidelines. The core EQuIS applications are its chemistry and geology modules, each of which is associated with its own underlying Microsoft SQL Server database. CH2M owns licenses for the geology and chemistry modules. The EQuIS database system is based on a relational model in which independent tables, each containing a certain type or entity of data, can be linked through selected fields that are common to two or more tables. The database design allows for the inclusion of historical data, and allows users to effectively conduct trend analysis and generate a variety of data reports to aid in data interpretation.

The database will be protected from unauthorized access, tampering, accidental deletions or additions, and data or program loss that can result from power outages or hardware failure. The following procedures will be adopted to ensure protection:

- The master database will be stored/hosted by EarthSoft, Inc., on a network file server with Web access from a local to the installation of the EQuIS data management system and access via EQuIS Enterprise Web interface and EQuIS Professional desktop software. Members of the data management team involved in loading, modifying, or querying the database will be given access through EQuIS user accounts and passwords, as well as the appropriate network server permissions.
- EQuIS Enterprise provides users with a Web-based interface and EQuIS Professional provides users with a desktop interface that allows for data reporting in standard formats. Where required, data exports from the master database will be stored on the local area network for access by project staff through custom reporting tools developed to minimize possible database corruption by users. Whenever the master database is updated or modified, the data will be recopied exported to the local area network to ensure that the current copy data set is available to users.
- EQuIS hosting takes place in secure, environmentally controlled, certified commercial data centers with controlled physical access. There dedicated environments provide: daily incremental backups, full weekly backups, backup generators, fire suppression, and 24-hour 7-day a week emergency restoration within 1 hour.

GIS Description

A project geodatabase will be agreed upon and set up prior to sampling by the SM, database manager, and GIS manager. Workflow for creating, maintaining, and organizing geospatial data will follow the Spatial Data Standard format for projects whenever possible. Sample location coordinates will be stored in both the Universal Transverse Mercator in meters and latitude/longitude in decimal degrees (both relative to the North American Datum of 1983).

An ArcView project or extension will be used providing the following functionality: load and display project site base maps; display sampling station locations and associated sampling data (date, media, and results); and perform ad hoc queries to highlight sampling locations meeting user-entered criteria for sampling (for example, data by date, sample type, analyte, depth/elevation, result value, or any combination thereof). Results will be shown as stations highlighted on the map.

Documentation

Documentation of data management activities is critical because it provides the following:

- An electronic copy record of project data management activities
- Reference information critical for database users
- Evidence that the activities have been properly planned, executed, and verified
- Continuity of data management operations when personnel changes occur

The data management plan will serve as the initial general documentation of the project data management efforts. Additional documentation will be maintained to document specific issues such as database structure definitions, database inventories, database maintenance, user requests, database issues and problems, and client contact.

Evidence File

The final evidence file will be the central repository for documents that constitute evidence relevant to sampling and analysis activities. CH2M is the custodian of the evidence file and maintains the contents of the evidence files for the project, including relevant records, reports, logs, field notebooks, sketches, pictures, contractor reports, and data reviews in a secured area with limited access.

CH2M will keep records until project completion and closeout. As necessary, records may be transferred to an offsite records storage facility. The records storage facility must provide secure, controlled-access records storage. Records of raw analytical laboratory data, QA data, and reports will be kept by the subcontracted laboratory for 5 years.

Presentation of Data

Depending on data user needs, data presentation may consist of the following formats:

- Tabulated results of data summaries or raw data
- Figures showing location-specific concentrations
- Tables providing statistical evaluation or calculation results
- Presentation tools, such as ARCINFO or similar analysis/presentation aids

In addition to laboratory data, other physical data will be collected during field efforts. The information will be stored in the project database. Other types of data elements may be added as the field investigation needs and activities evolve.

Assessment and Audit Tasks

See Worksheets #31, #32, and #33.

Data Review and Usability Tasks

A three-step data review process (consisting of verification, validation, and usability assessment) will be employed to examine the collected data so that only scientifically sound data of known and documented quality are used to make environmental decisions. Worksheets #34 through #37 (Data Verification Inputs, Data Verification Procedures, Data Validation Procedures, and Data Usability Assessment) describe the process and criteria in detail.

Environmental Services Assistance Team (ESAT) and CH2M will perform data validation on the analytical data obtained during the project according to the specifications provided in Worksheet #36 (Data Validation Procedures). Final data are placed in the database with qualifiers.

The data usability assessment is an evaluation based on the results of data validation in the context of the overall project decisions and objectives. The assessment is used to determine whether the project execution and resulting data meet the project DQOs (Worksheet #11). Both the sampling and analytical activities must be considered, with the ultimate goal of assessing whether the final, qualified results support the decisions to be made with the data. Worksheet #37 (Data Usability Assessment) describes the process in detail.

Additionally, the CH2M chemist will perform a 10 percent verification of the validated results against the EDD and hardcopy prior to data use.

Documentation and Records

Records and field measurements of samples will be collected in data sheets. Chains of custody, airbills, and sample logs will be prepared and retained for each sample.

A copy of the final QAPP will be kept at the CH2M St. Louis office.

Project Schedule

		Dates			
Activities	**Organization**	**Anticipated Date(s) of Initiation**	**Anticipated Date of Completion**	**Deliverable**	**Deliverable Due Date**
Site-specific plans	CH2M	March 2017	April 2017	Site-specific plans	April 6, 2017
Sample collection	CH2M	June 2017	August 2017	Samples to laboratory	NA
Laboratory analysis	TBD	Within required analytical holding times	October2017	Data package	60 days after receipt of samples
	ASL	Within required analytical holding times	September 2017	Data package	21 days after receipt of samples
Data validation	CH2M/ESAT	After receipt of analytical data reports	21 business days after receipt of final data	Data validation report	21 business days after receipt of final data
Data Evaluation Report	CH2M	After receipt of final data	January 2018	Date Evaluation Report	March 28, 2018
Work assignment completion report	CH2M	After receipt of the work assignment closeout notification	45 days after receipt of the work assignment closeout notification	Work assignment completion report	45 days after receipt of the work assignment closeout notification

NA = not applicable

TBD = to be determined

Worksheet #15—Project Action Limits and Laboratory-specific Detection/Quantitation Limits

One of the primary goals of the project-specific QAPP is to select appropriate analytical methods to achieve method detection limits (MDL) and reporting limits (RL) that will satisfy the overall project DQOs (as defined in Worksheets #10 [Conceptual Site Model] and #11 [Project/Data Quality Objectives]).

To determine whether the laboratory MDL and RL will meet the analytical DQOs, the MDLs and RLs are compared to the following project action limits (PALs) for the site:

- Preliminary Cleanup Goals (PRG) presented in the ROD, which were developed based on risk-based calculations (cancer and noncancer) and EPA Regional Screening Levels Residential Soil Criteria, June 2011

Table 15-1. Target Analytes, Methods, Action Levels, and Control Limits

Method	Chemical Group	Analyte	CAS Number	Units	Project Action Level (mg/Kg)	Quality Control Limits				
						MDL (mg/Kg)	RL (mg/Kg)	LCS/LCSD Recovery %	MS/MSD Recovery %	Relative Percent Difference (%)
ISM02.4	Metals	Arsenic	7440-38-2	mg/Kg	32	TBD	1	70-130	75-125	≤ 20
ISM02.4	Metals	Cadmium	7440-43-9	mg/Kg	37	TBD	0.5	70-130	75-125	≤ 20
ISM02.4	Metals	Lead	7439-92-1	mg/Kg	400	TBD	1	70-130	75-125	≤ 20
ISM02.4	Metals	Zinc	7440-66-6	mg/Kg	6,400	TBD	6	70-130	75-125	≤ 20
Laboratory SOP-02	Metals	Lead Bio-accessibility	7439-92-1	mg/Kg	NA	NA	200	80-120	80-120	≤ 20
SW6020A/ Laboratory SOP-03	Metals	Total Lead	7439-92-1	mg/kg	200	0.01	0.1	80-120	75-125	≤ 20

Notes:
NA = not applicable
MS/MSD = matrix spike/matrix spike duplicate

Worksheet #17—Sampling Design and Rationale

The site has 300 properties and 31 alleyways that require sampling to determine if remediation is required.

The properties are generally described as follows:

- 300 residential, commercial, and vacant properties not previously sampled or analyzed using XRF for arsenic, cadmium, zinc, and lead. It is assumed that each property will have up to 3 yard areas requiring sampling. All yard areas need to be sampled to identify if remediation is required, and the excavation depth, if required (assume each composite represent a 6-inch depth interval per yard area [0-6 inches, 6-12 inches, 12-18 inches, and 18-24 inches] from 600 yard areas (up to 3 yard areas per property) at 300 properties, for a total of 3,600 composite samples).
- 31 alleyways not previously sampled for arsenic, cadmium, zinc, and lead. It is assumed that each alley will require 16 composite samples to determine if remediation is required, and the excavation depth, if required (each composite representing a 2,500-square-foot (ft^2) area and 6-inch depth interval [0-6 inches, 6-12 inches, 12-18 inches, and 18-24 inches] from 31 alleyways, for a total of 496 samples).
- 30 residential properties not previously sampled for IVBA and total lead. It is assumed that one sample will be collected from the 0- to 1-inch interval at one yard area at each property, for a total of 30 samples.

Table 17-1 summarizes the property addresses and corresponding categories.

The field activities will be conducted according to the FOPs provided in Appendix A. The number of samples and the analytical parameters planned are summarized in Worksheet #18 (Sampling Locations and Methods).

17.1 Residential, Commercial, and Vacant Property Soil Sampling

- Composite soil sample collection at residential and commercial properties less than 5,000 ft^2: Up to three 5-point composite locations of soil will be collected (front, back, and side yards) at each proposed residential, commercial, or vacant property that is less than 5,000 ft^2. Samples will be collected from each of the 3 yard areas at 4 depth intervals: 06, 612, 1218, and 1824 inches below ground, resulting in a total of up to 12 composite soil samples at each property.
- Composite soil sample collection at residential and commercial properties greater than 5,000 ft^2: Up to four 5-point composites of soil will be collected (4 equally divided areas) at each proposed residential property that is greater than 5,000 ft^2. Samples will be collected from each of the 4 locations at 4 depth intervals: 06, 612, 1218, and 1824 inches below ground, resulting in a total of up to 16 composite soil samples at each property.

An estimated 12 composite samples will be collected from each of the 300 residential and commercial properties, for a total of up to 3,600 composite samples with an additional 720 QA/QC samples (4,320 total samples). Field QC sampling collection is summarized in Worksheet #20, and each field QC sample type is defined in Worksheet #28 of this QAPP. Field duplicates will be collected at a frequency of 1 per 10 field samples, and matrix spike (MS)/matrix spike duplicate (MSD) samples will be collected at a frequency of 1 pair per 20 field samples. One equipment blank per week (seven samples) will be collected for nondisposable or decontaminated equipment. Soil samples will be analyzed on a 60-day turnaround time for final results.

17.2 Alleyway Soil Sampling

- Composite soil sample collection at alleyways: Up to sixteen 5-point composite locations of soil will be collected at each alleyway. Each composite sample will represent a 2,500-ft^2 area and a 6-inch depth

interval: 06, 612, 1218, and 1824 inches below ground, resulting in a total of up to 16 composite soil samples at each property.

An estimated 16 composite samples will be collected from each of the 31 alleyways for a total of up to 496 composite samples with an additional 100 QA/QC samples (596 total samples). Field QC sampling collection is summarized in Worksheet #20, and each field QC sample type is defined in Worksheet #28 of this QAPP. Field duplicates will be collected at a frequency of 1 per 10 field samples, and MS/MSD samples will be collected at a frequency of 1 pair per 20 field samples. One equipment blank per week (1 sample) will be collected for nondisposable or decontaminated equipment. Soil samples will be analyzed on a 60-day turnaround time for final results.

17.3 Residential *In Vitro* Bioaccessibility Lead Sampling

- Composite soil sample collection at residential properties: One 30-point composite of soil will be collected from one yard (front, back, or side yard) at each proposed residential property.

An estimated 1 composite sample will be collected from each of the 30 yards for a total of up to 30 composite samples with an additional 7 QA/QC samples (37 total samples). Field QC sampling collection is summarized in Worksheet #20, and each field QC sample type is defined in Worksheet #28 of this QAPP. Field duplicates will be collected at a frequency of 1 per 10 field samples, and MS/MSD samples will be collected at a frequency of 1 pair per 20 field samples. One equipment blank per week (1 sample) will be collected for nondisposable or decontaminated equipment. Soil samples will be analyzed on a 21-day turnaround time for preliminary results.

17.4 Equipment Decontamination

Equipment decontamination specifics are detailed in SOP No. 8. Nondisposable equipment used during field activities will be decontaminated by washing with a phosphate-free detergent (for example, Liquinox), followed by a water rinse prior to reuse. Specifically, reusable sampling equipment, including the drill rods, hand augers and trowels, will be decontaminated after each use as follows:

1. Wear unpowdered chemical-resistant gloves.
2. Spray and scrub with a detergent solution.
3. Spray to rinse with distilled water.
4. Wipe dry with a clean paper towel.
5. Dispose of paper toweling and gloves in trash receptacles.

17.5 Investigative-Derived Waste Characterization and Disposal

Unless grossly contaminated, personal-protective equipment and disposable sampling equipment generated during the sampling will be disposed of in solid-waste receptacles rented for the project placed at a City- or County-owned property or at the FA, if access is granted. Soil cuttings will be placed back into the borings. Only minimal overspray of decontamination fluid to the alleyway and yards from decontamination of equipment will occur. It is assumed that no IDW will need to be managed for disposal.

Worksheet #18—Sampling Locations and Methods

Note: The approximate sample locations will be finalized once the GIS database is complete.

Figure 1, Site Plan and Existing Conditions, is provided on the following page.

Worksheets #19 and #30— Sample Containers, Preservation, and Hold Times

The analytical methods for each sample matrix, including the required sample volume, containers, preservation, and holding time requirements are provided in Table 19-1. Additional information on the laboratory analytical SOPs is provided in Worksheet #23 (Analytical SOP References).

Table 19-1. Sample Containers, Preservation, and Hold Times—CLP Laboratory (TBD)

Contact: TBD Email: TBD Phone: TBD	Certification: CLP Accreditation Expiration: NA Sample Delivery Method: FedEx Overnight services Data Deliverable: 60 Calendar Days
Back-up Laboratory:	TBD, if needed

Analyte Group	Matrix	Method/ SOP	Accreditation Expiration Date	Containers	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround Time
Metals	Soil	ISM02.4/ Laboratory SOP 1	NA	1 x 4-oz. glass jar	None	NA	180 days	60 days (final)

Worksheets #19 and #30—Sample Containers, Preservation, and Hold Times (Continued)

Applied Sciences Laboratory
Kathy McKinley
1100 NE Circle Boulevard, Suite 300
Corvallis, Oregon 97330
E-mail: Kathy.McKinley@ch2m.com
Phone: (541) 768-3144

Back-up Laboratory:

Sample Delivery Method:

Analyte Group	Matrix	Method/ SOP	Containers	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround Time
IVBA	Soil	Physiologically Based Extraction (bio-accessibility of lead)/Laboratory SOP-02	1 x 4-oz. glass jar	< 6 degrees Celsius	NA	180 days	21 days (final)
Total Lead	Soil	SW6020A/SOP-03					

NA = not applicable
One sample container will be used for both IVBA and total lead analysis

Worksheet #20—Field Quality Control Summary

20.1 Field Duplicate Samples

Field duplicates are two field samples taken concurrently at the same location. They are intended to represent the same population and are taken through all steps of the sampling and analytical procedures in the same manner as the associated native sample. The samples are used to assess the precision of the entire data collection activity, including sampling, sample handling and storage, and site heterogeneity. The field duplicates are assigned a unique sample name and are collected in a separate container from the associated native sample. One field duplicate will be collected for every 10 field samples.

20.2 Matrix Spike/Duplicate Samples

MS/MSD samples are an aliquot of the sample spiked with known concentrations of specific analytes. The spiking occurs before sample preparation and analysis at the laboratory. To allow the analytical laboratory to run MS/MSD and/or MS/duplicate analyses, additional sample volumes will be collected in the field to provide sufficient sample volume. An MS/MSD pair will be collected for every 20 field samples for IVBA, and an MS/duplicate pair will be collected for every 20 field samples analyzed for metals.

20.3 Blanks

An equipment blank is a sample of laboratory-grade deionized water poured into, over, or pumped through the sampling equipment, collected in the appropriate sample container, and analyzed by the laboratory for the same parameters as the field samples. These blanks are used to assess the effectiveness of equipment decontamination procedures. One equipment blank per week will be collected for nondisposable and decontaminated equipment.

Worksheet #20—Field Quality Control Summary Table

Matrix	Analyte/ Analytical Group	Estimated Number of Field Samples	Field Duplicates	Matrix Spikes/ Matrix Spike Duplicates [a]	Field Blanks	Equipment Blanks	Trip Blanks	Other	Estimated Total No. Analyses
Surface, Subsurface Soils (Residential/ Commercial/Vacant)	Total Lead (soil)	3,600	360	180/180	0	7	0	0	4,327
Surface, Subsurface Soils (Residential/ Commercial/Vacant)	Total Arsenic (soil)	3,600	360	180/180	0	7	0	0	4,327
Surface, Subsurface Soils (Residential/ Commercial/Vacant)	Total Zinc (soil)	3,600	360	180/180	0	7	0	0	4,327
Surface, Subsurface Soils (Residential/ Commercial/Vacant)	Total Cadmium (soil)	3,600	360	180/180	0	7	0	0	4,327
Surface, Subsurface Soils (Alleyways)	Total Lead (soil)	496	50	25/25	0	1	0	0	597
Surface, Subsurface Soils (Alleyways)	Total Arsenic (soil)	496	50	25/25	0	1	0	0	597
Surface, Subsurface Soils (Alleyways)	Total Zinc (soil)	496	50	25/25	0	1	0	0	597
Surface, Subsurface Soils (Alleyways)	Total Cadmium (soil)	496	50	25/25	0	1	0	0	597
Soil	Total Lead (soil)	30	3	2/2	0	1	0	0	38
Soil	IVBA	30	3	2/2	0	1	0	0	38

Worksheet #21—Standard Field Operating Procedures

Reference Number	Title, Revision, Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Check if yes)	Comments
FOP-01	Soil sampling in Residential, Commercial and Vacant Areas	CH2M	Procedural guidance	☐	
FOP-02	Soil sampling in Alleyways	CH2M	Procedural guidance	☐	
FOP-03	*In Vitro* Bioaccessibility sampling in Residential Areas	CH2M	Procedural guidance	☐	
FOP-04	Utility Clearance for Intrusive Operations	CH2M	Procedural guidance	☐	
FOP-05	Sample Handling and Chain-of-Custody Procedure	CH2M	Procedural guidance	☐	
FOP-06	Packing and Shipping of Environmental Samples	CH2M	Procedural guidance	☐	
FOP-07	Note Taking and Field Logbook	CH2M	Procedural guidance	☐	
FOP-08	Equipment Decontamination Procedures	CH2M	Procedural guidance	☐	

Worksheet #22—Field Equipment Calibration, Maintenance, Testing, and Inspection

☑ Worksheet Not Applicable (State Reason)

No instruments or equipment requiring calibration, maintenance, testing, or inspection are required for this project. In the event a specific task requires this, the instrument and equipment maintenance, testing, and inspection activities are documented per the FOPs listed in Worksheet #21.

Worksheet #23—Analytical SOPs

The following laboratory SOP references have not been modified for this project and may not reflect the exact requirements of this document. The laboratory SOPs are supplemented by internal communications within the laboratory to disseminated the project requirements to technical staff. Laboratory SOPs are available upon request.

Reference Number	Title, Revision, Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
SOP-01	Superfund Methods Multi-media, Multi-concentration, ISM02.4, October 2016	Definitive	Metals	ICP-AES	Contract Program Laboratory (CLP)	N
SOP-02	Physiologically Based Extraction Procedure (PBEP) MET15.0, Rev. 4, January 2015	Definitive	IVBA	NA	Applied Sciences Laboratories[a]	Y
SOP-03	Standard Operating Procedures for the Determination of Metals by ICP-MS, Rev. 12, August 2015	Definitive	IVBA/total lead	ICP-MS	Applied Sciences Laboratories[a]	N
SOP-04	Standard Operating Procedure for Hot Block Digestion of Sediment and Soils, Rev. 10, February 2015	Definitive	Total Lead	Hot Block	Applied Sciences Laboratories[a]	N

[a] Chicago Regional Laboratory (CRL) is the secondary laboratory. CRL is in the process of adding these parameters and will keep in-house if pass certification.

Worksheet #24—Analytical Instrument Calibration

To confirm that the analytical methods and the selected instrumentation meet the project requirements, each analytical instrument will be calibrated according to the procedures outlined in the tables provided in Worksheet #28 (Analytical Quality Control and Corrective Action). Worksheets #24 and #28 have been combined for efficiency and ease of use to the CH2M project chemist and the laboratory. The information provides documentation on corrective actions, flagging criteria for laboratory services, and expectations for analytical services. Tables meet the requirements of both Worksheet #28 (Analytical Quality Control and Corrective Action) and Worksheet #24 (Analytical Instrument Calibration). Tables are presented by method and reflect the individual method requirements.

Worksheet #25—Analytical Instrument and Equipment Maintenance, Testing, and Inspection

☑ Worksheet Not Applicable (State Reason)

The laboratories will keep all maintenance, testing, and inspection records on file at the laboratory. The instrument and equipment maintenance, testing, and inspection activities are documented per the SOPs listed in Worksheet #23.

Worksheets #26 and #27—Sample Handling, Custody, and Disposal

Sample Collection, Packaging, and Shipment

Sample Collection (Personnel/Organization): Field team leader/CH2M

Sample Packaging (Personnel/Organization): Field team leader/CH2M

Coordination of Shipment (Personnel/Organization): Field team leader/CH2M

Type of Shipment/Carrier: Federal Express Overnight

Sample Receipt and Analysis

Sample Receipt (Personnel/Organization): Laboratory Personnel

Sample Custody and Storage (Personnel/Organization): Laboratory Personnel

Sample Preparation (Personnel/Organization): Laboratory Personnel

Sample Determinative Analysis (Personnel/Organization): Laboratory Personnel

Sample Archiving

Field Sample Storage (No. of days from sample collection): See QAPP Worksheet #19 for allowable holding time. The laboratory shall retain samples for at least 90 days after receipt.

Sample Extract/Digestate Storage (No. of days from extraction/digestion): See QAPP Worksheet #19 for allowable holding time.
The laboratory sample custodian will store all extracts/digestates for 30 days after final report has been submitted.

Biological Sample Storage (No. of days from sample collection): Not applicable.

Sample Disposal

Personnel/Organization: Laboratory Personnel

Number of Days from Analysis: The laboratory will retain samples for at least 90 days and sample extracts for at least 30 days, after submittal, pending the need for reanalysis.

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory)

Sample handling and chain-of-custody procedures will be performed per FOP-05, and packaging and shipping of environmental samples per FOP-06.

Sample coolers will be shipped to arrive at the laboratory the morning after sampling (priority overnight) or will be sent by a courier to arrive the same day. The laboratory will be notified of the sample shipment and the estimated date of arrival of the samples being delivered.

Regulations for packaging, marking/labeling, and shipping of hazardous materials and wastes are promulgated by the U.S. Department of Transportation. Air carriers that transport hazardous materials, in particular FedEx, require compliance with the current edition of the International Air Transport Association Dangerous Goods Regulations, which applies to shipment and transportation of hazardous materials by air carrier. Following current International Air Transport Association regulations will ensure compliance with U.S. Department of Transportation regulations**.**

Laboratory Sample Custody Procedures (receipt of samples, archiving, and disposal)

Upon sample receipt, the laboratory sample custodian will verify package seals, open the packages, check temperature blanks, record temperatures, verify sample integrity, and inspect contents against chain-of-custody forms. The project chemist will be contacted to seek resolution of any discrepancies between sample containers and chain-of-custody forms through contract-defined channels of communication. Once the shipment and chain-of-custody form are in agreement, the sample custodian will initiate an internal chain-of-custody form, as well as supply the laboratory task manager with a sample acknowledgement letter. When applicable, sample preservation will be checked and pH will be documented. If the sample temperatures are outside the required range, the laboratory will contact the project chemist as to the proper course of action.

Samples will be logged in and assigned a unique laboratory number for each sample, and the number will be used by all laboratory personnel handling samples to ensure all sample information is captured. Analyses required will be specified by codes assigned to samples at login. Labels containing the laboratory sample number are generated and placed on sample bottles.

After the laboratory labels the samples, they will be moved to refrigerators where they will be maintained at less than 6 degrees Celsius.

When the analyst is ready to prepare and/or analyze the sample(s), an appropriate member of the sample management department will locate the sample(s) in the locked refrigerator, sign and date the internal sample tracking form, and provide the sample(s) to the analyst. When the analyst is finished with the sample(s), unused portions will be returned to an appropriate member of the sample management department for replacement in a secure refrigerator. The analyst will sign and date internal chain-of-custody forms. In the event that entire samples are depleted during analysis, a notation of “sample depleted” or “entire sample used” will be written on the internal chain-of-custody forms.

Samples will be stored in designated secure, refrigerated storage areas. Samples and sample extracts will be maintained in a secure storage until disposal. No samples or extracts will be disposed of without prior written approval from an appropriate member of the project team. The sample custodian will note the sample disposal date in the sample ledger. The laboratory will dispose of samples in accordance with applicable regulations.

Documentation will be placed in a single, secured project file maintained by the laboratory project manager. The file will consist of the following components: agreements, correspondence, memorandums, notes, and data.

Reports (including QA reports) will be filed with correspondence. Analytical laboratory documentation, field data, and notes will be filed with the laboratory data. Filed materials may only be removed by authorized personnel on a temporary basis. The name of the person removing the file will be recorded. Laboratories will retain project files and data packages for 7 years, unless otherwise agreed.

Sample Identification Procedures

A sample numbering system will be used to identify each sample, including duplicate and replicate samples. The sample number will be a unique identifier.

Each sample, regardless of analytical protocol, will also be assigned a CH2M site-specific identifier, which will contain a site- and sample-specific location identifier that indicates where the sample was obtained.

The sample number and station location identifier will be included on the sample tag, the traffic report, and the chain-of-custody record.

The site-specific identifier is based on the following system:

Residential, Commercial, and Vacant Soil Samples

Station IDs will be assigned to each address prior to the field event (i.e., a number 001 through 1,000). The sample ID will consist of the site identifier, station ID, the yard designation, and the depth interval.

- **Site Identifier—**The site is Old American Zinc Plant Superfund Site and is designated with a 3-digit ID: OAZ.
- **Station Location—**The station location identifier is the 3-digit unique ID. The station location IDs will be assigned to each property where access has been obtained and will be provided in a table showing the station location ID (001 through 1000) and the property address.
- **Yard Designation—**The yard designation identifier follows the station location identifier. The yard designation will provide the area of the yard where the sample was collected. For properties less than 5,000 square feet, the yard designations are “F” for front yard, “B” for back yard, or “S” for side yard. For properties greater than 5,000 square feet, the yard designations are “A”, “B”, “C”, and “D” when the yard is divided into parallel sections.
- **Depth Indicator—**Depth indicator codes will follow the yard designation. The code will consist of a hyphen, followed by the starting and bottom depth intervals separated by a slash. The indicator will provide the depth that represents the start and end of the sample interval in inches below ground. For example, the sample depth designation will be “-06” for the sample collected from an interval of 0 to 6 inches below ground. A soil sample from the front yard of property 156 taken from the depth of 0 to 6 inches would be identified as OAZ-156F-00/06.

Other Samples

- **QA/QC Identifier—**Field QA/QC samples will be identified using the following identifiers:
  - Equipment blanks, which are not associated with an individual station location, are numbered sequentially and are identified by the first two letters of the station location code (for example, EB001).

  - Field duplicates, which are associated with the same station location as the native sample, are identified with an "R" (for "replicate") appended to the end of the location code. For example, the duplicate of sample OAZ-156F-00/06 would be labeled OAZ-156F-00/06R.

- **Laboratory QC Samples—**A sample collected for laboratory QC, such as a matrix spike sample, is considered to be a single sample, even though additional volume is provided to the laboratory. Laboratory QC samples are assigned a single sample number and station location identifier. Laboratory QC samples are not identified in the station location code but rather are called out on the chain-of-custody form in the *Samples to be used for laboratory QC* field and on the sample tag.

- **IVBA Samples—**Composite samples collected for the IVBA study will be identified with the site identifier, station location ID, the yard designation and the depth interval. A sample collected from the front yard of property 156 taken from a depth interval of 0 to 1 inches would be identified as OAZ-156F-00/01.

- **Alleyway Soil Samples—**Station IDs will be assigned to each alleyway prior to the field event (i.e., a number A01 through A31). The sample ID will consist of the site identifier, station ID, the composite designation, and the depth interval. The composite designation will be assigned a two-digit number (i.e., a number 01 through 99) for the section of the alleyway and the numbering scheme will start North to South or East to West (depending on the direction of the alleyway). A sample collected from the furthest East section of the A02 alleyway taken from a depth interval of 6 to 12 inches would be identified as OAZ-A02-01-06/12.

Chain-of-Custody Procedures

Chains of custody will include, at a minimum, laboratory contact information, client contact information, sample information, and relinquished by/received by information in accordance with the SOP. Sample information will include sample identification, date and time collected, number and type of containers, preservative information, analysis method, and comments. The chain of custody will also have the sampler's name and signature. The chain of custody will link location of the sample from the field logbook and sample processing log through sample disposal by the laboratory. See Appendix A, FOP-05, Attachment 1, for an example chain-of-custody form. The laboratory will use the sample information to populate the laboratory database for each sample.

Worksheet #28—Analytical Quality Control and Corrective Action

Worksheet #28 presents analytical QC requirements relevant to the analysis of environmental samples that the laboratories will follow to produce definitive data. The purpose of the laboratory QC sample activities is to produce data of known quality that satisfy the project-specific DQOs. Laboratory QC samples will follow the analytical methods and are presented in Tables 28-1 through 28-2. The type of laboratory QC samples and the frequency of use of these samples are discussed below and in method-specific Laboratory SOPs.

Analytical Quality Control

Calibration

Analytes reported must be present in the initial and continuing calibrations. The calibrations must meet the acceptance criteria specified in the tables provided in this QAPP. Results reported must be within the calibration range. Samples will be diluted, if necessary, to bring analyte responses within the calibration range. Records of standard preparation and instrument calibration will be maintained. Records must unambiguously trace the standards and their use in calibration and quantitation of sample results.

Instrument calibration will be performed by beginning with the simplest approach first, the linear model through the origin, and then progressing through other options until the acceptance criteria are met. In cases where an analyte has more than one acceptable calibration model, results from the simplest calibration model will be reported. If more than the minimum number of standards is analyzed for the initial calibration (ICAL), the standards analyzed will be included in the ICAL. The only exception to this rule is that a standard at either end of the calibration curve can be dropped from the calibration, providing that the requirement for the minimum number of standards is met and the low point of the calibration curve is at or below the RL for each analyte.

Calibrations must use the simplest calibration model first. Non-linear calibration will be considered only when a linear approach cannot be applied. It is not acceptable to use an alternate calibration procedure when a compound fails to perform in the usual manner. When this occurs, it is indicative of instrument issues or operator error.

The continuing calibration verification (CCV) cannot be used as the laboratory control sample (LCS), except for methods that do not involve sample preparation. A CCV will be performed daily before sample analysis (unless an ICAL and second-source standard verification is performed immediately before sample analysis) and as required by the applicable method. Finally, the lowest standard used must be at or below the reporting for each analyte in the method.

Method Blank

For method blanks, the source of contamination will be investigated. If one-half the RL for any analyte (or the RL for common laboratory contaminants) is exceeded, the laboratory will evaluate whether the samples need to be reprocessed using the following criteria: (1) the method blank contamination exceeds a concentration greater than one-tenth of the measured concentration of any sample in the associated preparation batch, or (2) there is evidence that the blank contamination otherwise affects the sample results. Except when sample analysis results in an ND, samples associated with method blank

contamination and meeting these criteria will be reprocessed in a subsequent preparation batch. If insufficient sample volume remains for reprocessing, the results will be reported with a B-flag, along with any other appropriate data qualifier. If an analyte is detected only in the method blank, and not in any batch samples, no flagging is necessary. Method blank contamination must be addressed in the case narrative.

Laboratory Control Sample

An LCS is a sample of known composition that is spiked with target analytes. The LCS is used with each analytical batch to determine whether the method is in control. Each analyte in the LCS will be spiked at a level less than or equal to the midpoint of the calibration curve, which is defined as the median point of the curve instead of the middle of the range. The LCS will be carried through the complete sample preparation and analysis procedure. The LCS cannot be used as the CCV.

At least one LCS will be included in every analytical batch. If more than one LCS is analyzed in an analytical batch, results from LCSs will be reported. A failure of an analyte in any of the LCSs will require appropriate corrective action, including qualification of the failed analyte in the samples, as required.

Laboratory Control Limits

The LCS control limits (CLs) are specified in Worksheet #15. The performance of the LCS is evaluated against the QC acceptance limits. Whenever an analyte in the LCS is outside the acceptance limit, corrective action will be performed.

Marginal Exceedance

Sporadic marginal exceedances (MEs) are not allowed for target analytes without project-specific approval. MEs are for those analytes outside CLs (three standard deviations) but still within four standard deviations.

MEs must be sporadic (random). If the same analyte exceeds the LCS CLs repeatedly (e.g., two out of three consecutive LCSs), that is an indication that the problem is systematic, not random. The source of systematic error should be located, and appropriate corrective actions should be taken.

Matrix Spike/Matrix Spike Duplicate

An MS or MSD is an aliquot of sample collected in the field and spiked with known concentrations of target analytes. The spiking occurs before sample preparation and analysis. Each analyte in the MS and MSD will be spiked at a level less than or equal to the midpoint of the calibration curve for that analyte. The MS/MSD is used to document potential matrix effects associated with a site and will not be used to control the analytical process. The MS/MSD results and flags will not be associated with or related to samples that are collected from the same site where the MS/MSD set was collected, with the exception of an FD. Additional volume will be collected for samples selected for MS/MSDs, and the laboratory will use those samples to prepare the appropriate MS/MSDs.

The performance of the MS and MSD is evaluated against the QC acceptance limits outlined in Worksheet #15. If either the MS or the MSD is outside the QC acceptance limits, the data will be evaluated to determine whether there is a matrix effect or analytical error. The analytes in the parent sample and associated FD collected at the same site (if applicable) will be qualified according to the data flagging criteria of this QAPP.

If the sample concentration exceeds the spike concentration by a factor of four or more, the data will be reported unflagged. The laboratory should communicate potential matrix difficulties to the CH2M project chemist so an evaluation can be made with respect to the project-specific DQOs.

Quality Control Checks

Analytical Batch Requirements

Laboratory QC samples will be included in an analytical batch with the field samples. An analytical batch is a group of samples (not exceeding 20 environmental samples plus associated laboratory QC samples) that are similar in composition (matrix), extracted or digested at the same time and with the same lot of reagents, and analyzed together as a group.

The analytical batch also extends to cover samples that do not need separate extraction or digestion (e.g., volatile organic compound analysis by purge and trap). The identity of each analytical batch will be unambiguously reported with the analyses so that a reviewer can identify the laboratory QC samples and the associated environmental samples. The type of laboratory QC samples and the frequency of use of these samples are discussed in the following sections.

Holding-Time Compliance

Sample preparation and analysis will be completed within the method required holding times, as noted in Worksheets #19 and #30 (Sample Containers, Preservation, and Holding Times). Some methods have more than one holding time requirement (e.g., Methods SW8081A and SW8270D). For methods not requiring sample preparation, the holding time is calculated from the time of sample collection to the time of completion of analytical runs. The following apply to methods requiring sample preparation before analysis:

- Preparation holding time is calculated from the time of sample collection to the time of completion of preparation.
- Analytical holding time is calculated from the time of completion of preparation to the time of completion of analytical runs.

Holding times are determined on the basis of days, hours, and minutes. If the time of sample collection is not provided, the laboratory must assume the most conservative time of day. If holding times are exceeded and the analyses are performed, the results will be flagged and identified in the data package case narrative.

Control Charts

Control charts or data analysis software will be maintained and used to detect trends and prevent out-of-control conditions. CLs will be monitored on an ongoing basis (at least quarterly) for shifts in mean recovery, changes in standard deviation, and development of trends. Laboratories may choose representative compounds for control charts for the purpose of trend analysis.

The laboratory QA Officer or designee will review control charts at a specified frequency for out-of-control conditions and initiate appropriate corrective actions. Data analysis software may also be used for the statistical evaluation of data for trends and biases.

Standard Materials

Standard materials including second source materials used in calibration and to prepare samples will be traceable to a National Institute of Standards and Technology (NIST), EPA, American Association of Laboratory Accreditation (A2LA), or other equivalent approved source, if available. If a NIST, EPA, or A2LA standard material is not available, the standard material proposed for use will be included in an addendum to the project-specific QAPP and approved before use.

The standard materials will be current, and the following expiration policy will be followed:

- Expiration dates for ampulated solutions will not exceed the manufacturers' expiration date or 1 year from the date of receipt, whichever comes first.
- Expiration dates for laboratory-prepared stock and diluted standards will be no later than the expiration date of the stock solution or material or the date calculated from the holding time allowed by the applicable analytical method, whichever comes first.
- Expiration dates for pure chemicals will be established by the laboratory and be based on chemical stability, possibility of contamination, and environmental and storage conditions.

Expired standard materials will be either revalidated before use or discarded. Revalidation may be performed through assignment of a true value and error window statistically derived from replicate analyses of the material as compared to an unexpired standard. The laboratory will label standard and QC materials with expiration dates.

A second source standard is used to independently confirm the ICAL. A second source standard is a standard purchased from a vendor different from that supplying the material used in the ICAL. The second source material can be used for the continuing calibration standards and/or for the LCS. Two different lot numbers from the same vendor do not normally constitute a second source. However, when a project requires analyses for which there is not a separate vendor source available, the use of different lot numbers from the same vendor will be acceptable to verify calibration.

Supplies and Consumables

The laboratory will inspect supplies and consumables before their use in analysis. The materials description in the methods of analysis will be used as a guideline for establishing the acceptance criteria for these materials. Purity of reagents will be monitored and documented. An inventory and storage system for these materials will ensure that the materials are used before manufacturers' expiration dates and are stored under safe and chemically compatible conditions.

Worksheet #28—Analytical Quality Control and Corrective Action

Table 28-1

Matrix Soils

Analytical Group Metals (arsenic, lead, zinc, cadmium)

Analytical Method/SOP ISM02.4/SOP 1

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
Linear Dynamic Range (LDR) or High level check standard	At initial set-up and checked every 6 months with a high standard at the upper limit of the range.	Within ±10% of true value.	Dilute samples within the calibration range, or re-establish/verify the LDR.	Not appropriate.
ICAL (at least six calibration standards)	Daily ICAL prior to sample analysis.	Correlation coefficient ≥ 0.995. Percent difference (%D) for each standard ± 30%	Correct problem, then repeat ICAL.	Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.
ICV/Second-source calibration verification	Once per ICAL prior to sample analysis.	All analytes within ± 10% of expected value.	Correct problem and verify second-source standard. Rerun second-source verification. If that fails, correct problem and repeat ICAL.	Problem must be corrected. Samples may not be analyzed until the calibration has been verified.
CCV	At the beginning of the analytical sequence, every two hours and at the end of the analysis sequence.	All analytes within ± 10% D of expected value of true value.	Recalibrate and reanalyze all affected samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	Within ± 20% of true value or within ±1x CRQL true value whichever is greater.	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all samples.	If corrective action fails, flag all results for specific analyte(s) in all samples associated with the failed ICS.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected >CRQL.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.
Method Blank	One per extraction batch.	No analytes ≥CRQL.	Investigate source of contamination. Reanalyze blank and associated samples.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative.

Table 28-1

Matrix Soils

Analytical Group Metals (arsenic, lead, zinc, cadmium)

Analytical Method/SOP ISM02.4/SOP 1

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	One per 20 field samples.	Acceptance Criteria: Worksheet #15.	If the MS is outside recovery limits, qualify results. Only applicable to sample concentrations <4x the spike concentration.	Flag samples received and associated with the spike sample "*"
Serial Dilution (ICP Analysis Only)	One per Analytical Batch per matrix. Field blanks cannot be used serial dilution analysis.	Concentration of reported analytes are > 50 times the MDL and RPD ≤10%.	Correct problem, then re-prepare and analyze the SD and all samples in the affected analytical batch.	Flag samples associated with the sample "*"
Post-Digestion Spike	Upon failure of MS/MSD, applicable to sample results <4x spike.	75-125%	Qualify for matrix interference.	Flag samples received and associated with the spike sample "*"
Laboratory Control Sample (LCS)	One per analytical batch.	Acceptance Criteria: Worksheet #15.	Correct problem, then re-prepare and analyze the LCS and all samples in the affected analytical batch.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative.
Laboratory Duplicate	1 per analytical batch.	RPD ≤ 20%	Reanalyze sample and duplicate. If still outside control limits, report to client and narrate.	Flagging not appropriate.

Worksheet #28—Analytical Quality Control and Corrective Action

Table 28-2

Matrix Soils

Analytical Group Metals

Analytical Method/SOP Physiologically Based Extraction (bio-accessibility of lead)/ SOP 2; SW6010C/ SOP 3

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
Method Blank	One per extraction/analytical batch.	No analytes ≥CRQL	Investigate source of contamination. Reanalyze blank and associated samples.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative.
LCS/LCSD	One per extraction/analytical batch.	Acceptance Criteria: Worksheet #15	Correct problem, then re-prepare and analyze the LCS and all samples in the affected analytical batch.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative.
MS/MSD	One per 20 samples.	Acceptance Criteria: Worksheet #15	Assess data to determine whether there is a matrix effect or analytical error. Analyze LCS for failed target analytes. Potential matrix effects should be communicated to CH2M so an evaluation can be made regarding the DQOs.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative.
Laboratory Duplicate	One per 20 samples if MS/MSD not requested.	RPD ≤ 20%	Reanalyze sample and duplicate. If still outside control limits, report to client and narrate.	Flagging not appropriate
Linear Dynamic Range (LDR) or High-level Check Standard	At initial set-up and checked every 6 months with a high standard at the upper limit of the range.	Within ±10% of true value.	Dilute samples within the calibration range, or re-establish/verify the LDR.	Not appropriate.

Table 28-2

Matrix Soils

Analytical Group Metals

Analytical Method/SOP Physiologically Based Extraction (bio-accessibility of lead)/ SOP 2; SW6010C/ SOP 3

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
ICAL (Minimum one high standard and a calibration blank.	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r2 ≥ 0.99.	Correct problem, then repeat ICAL.	Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.
ICV/Second-source calibration verification	Once per ICAL prior to sample analysis.	All analytes within ± 10% of expected value.	Correct problem and verify second-source standard. Rerun second-source verification. If that fails, correct problem and repeat ICAL.	Problem must be corrected. Samples may not be analyzed until the calibration has been verified.
CCV	After every 10 field samples and at the end of the analysis sequence.	All analytes within ± 10% D of expected value of true value.	Recalibrate and reanalyze all affected samples since last acceptable CCV	If reanalysis cannot be performed, data must be qualified and explained in the case narrative.
Low Level Calibration Check Standard (low-level ICV)	Daily.	All analytes within ± 20% D of expected value of true value.	Correct problem and repeat ICAL	Flagging not appropriate. No samples will be analyzed without a valid low-level calibration check standard.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected >LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.

Table 28-2

Matrix Soils

Analytical Group Metals

Analytical Method/SOP Physiologically Based Extraction (bio-accessibility of lead)/ SOP 2; SW6010C/ SOP 3

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all no spiked project analytes < LOD (unless they are verified trace impurity from one of the spiked analytes). ICS-AB: Within ± 20% of true value.	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all samples.	If corrective action fails, qualify all results for specific analyte(s) in all samples associated with the failed ICS.
Dilution Test Only applicable for samples with concentrations > 50 X CRQL (prior to dilution). Use along with MS/MSD or Post -Digestion Spike data to confirm matrix effects.	Once per batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	N/A	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.
Post-Digestion Spike Addition Criteria apply for samples with concentrations < 50 X CRQL prior to dilution.	One per batch if MS or MSD fails (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%	N/A	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.

Worksheet #29—Project Documents and Records

Sample Collection Documents and Records	Onsite Analysis Documents and Records	Offsite Analysis Documents and Records	Data Assessment Documents and Records	Other
Field notes Scribe database Chain-of-custody records Air bills Custody seals Telephone logs Corrective action forms Property sketches	Sample receipt, custody, and tracking records Equipment calibration logs Equipment maintenance, testing, and inspection logs Corrective action forms Reported field sample results Telephone logs Analysis logs	Sample receipt, custody, and tracking records Narrative Standard traceability logs Equipment calibration logs Sample preparation logs Run logs Equipment maintenance, testing, and inspection logs Corrective action forms Reported field sample results Reported results for standards, QC checks, and QC samples Instrument printout (raw data) for field samples, standards, QC checks, and QC samples Data package completeness checklists Extraction/cleanup records Raw data (stored electronically) QA review records	Data verification/validation reports Corrective action forms	Project database for analytical and field data Health and saftey briefing information Staff health and safety records

The laboratory data package will be organized such that the analytical results are reported on a per analytical batch basis, unless otherwise specified. In addition to the summary data deliverable, a full-supporting raw data deliverable package is required from the laboratory. All data will be provided electronically as a PDF file. CH2M will provide data copies to EPA.

An EDD is also required for all data. The laboratory will provide CH2M with an EDD in the current EQuIS format. The data will undergo QA reviews prior to being loaded to the project database. Delivery time for data from the laboratory will vary based on project-specific data use.

Sample Tracking Program

The EPA Scribe program will be used for field documentation and generation of chains of custody. Refer to EPA Office of Solid Waste and Emergency Response 9240.0-44, EPA 540-R-07-06 Contract Laboratory Program Guidance for Field Samplers, dated July 2007.

Project-specific Deliverables

Analytical data will be exported into a format consistent with the EDD format specified by EPA Region 5. Data will be submitted to EPA Region 5 in accordance with the requirements located here: https://www.epa.gov/sites/production/files/2017-01/documents/r5comprehensivemanual_jan2017.pdf.

Documentation and reports specified in this QAPP will be retained in Adobe PDF format.

Worksheets #31, #32, and #33—Assessments and Correction Action

Assessments:

Assessment Type	Responsible Party & Organization	Number/ Frequency	Estimated Dates	Assessment Deliverable	Deliverable due date
Data Review and Verification	Shane Lowe, Project Chemist/CH2M Quality Assurance Officer (QAO)/Laboratory TBD	All data packages	TBD	Email of deficiencies	After arrival of data from the laboratory and during data verification activities
Field Progress Report	TBD, Sampling Manager, Field Team Leader/CH2M, Field Quality Manager/CH2M	Daily	TBD	Daily Report	Daily during soil sampling field activities
Data Validation	ESAT and CH2M	After receiving data from laboratory	TBD	Data Validation report	21 business days after receipt of validated data
Data Quality Evaluation	Shane Lowe, Project Chemist/CH2M	One report after all data are validated	TBD	Data Quality Evaluation report	45 business days after project completion

Assessments and Corrective Action:

Assessment Type	Responsibility for responding to assessment findings	Assessment Response Documentation	Timeframe for Response	Responsibility for Implementing Corrective Action	Responsible for monitoring Corrective Action implementation
Daily field documentation reviews	TBD, Sampling Manager and Field Team Leader/CH2M, Field Quality Manager/CH2M	Field notes/ daily email	As soon as notification of corrective action is received	TBD, Sampling Manager and Field Team Leader/CH2M, TBD, Field Quality Manager/CH2M	Rachel Grand, CH2M SM
Data review and verification	Shane Lowe, Project Chemist/CH2M, QAO/Laboratory TBD	Corrective action reports and/or updated case narratives and corrected data submissions	3 to 5 business days	QAO/Laboratory TBD	Shane Lowe, Project Chemist/CH2M

Worksheet #34—Data Verification and Validation Inputs

To ensure that scientifically sound data of known and documented quality are used in making environmental decisions, the following three-step data review will be performed. Step I (verification) will confirm that all specified activities involved in collecting and analyzing samples have been completed and documented and that the necessary records (objective evidence) are available to proceed to data validation. Step II (validation) will assess whether the sampling and analytical processes comply with the contract-specific and the QAPP-specific requirements. Step III (usability assessment) will determine whether the resulting data are suitable as a basis for the decision being made. Worksheets #34 to #37 describe the processes to be followed. Worksheet #34 establishes the procedures that will be followed to verify project data including, but not limited to, sampling documents and analytical data package. The items subject to verification and validation are listed in the following table.

Item	Description	Verification (completeness)	Validation (conformance to specifications)
Planning Documents/Records			
1	Approved QAPP	X	
2	SOPs	X	
3	Laboratory SOPs	X	
Field Records			
4	Field logbooks	X	X
5	Field Forms	X	X
6	Chain-of-custody forms	X	X
Analytical Data Package			
7	Cover sheet (laboratory identifying information)	X	X
8	Case narrative	X	X
9	Internal laboratory chain-of-custody	X	X
10	Sample receipt records	X	X
11	Sample chronology (that is, dates and times of receipt, preparation, and analysis)	X	X
12	RL/MDL establishment and verification	X	X
13	Standards traceability	X	X
14	Instrument calibration records	X	X
15	Definition of laboratory qualifiers	X	X
16	Results reporting forms	X	X
17	QC sample results	X	X
18	Corrective action reports	X	X
19	Electronic data deliverable	X	X

Worksheet #35—Data Verification Procedures

Data verification is a completeness check to confirm that all required activities were conducted, all specified records are present, and the contents of the records are complete. It applies to both field and laboratory records.

Table 35-1.

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Field Notes and Data Sheets	Verify that records are present and complete for each day of field activities. Verify boring logs. Verify that all planned samples, including field QC samples were collected and that sample collection locations are documented. Verify that meteorological data were provided for each day of field activities. Verify that changes/exceptions are documented and were reported in accordance with requirements.	Internal	TBD, Sampling Manager and Field Team Leader/CH2M TBD, Field Quality Manager/CH2M Rachel Grand, SM/CH2M
Field SOPs	Verify that the sampling SOPs were followed.	Internal	Field Team Leader/CH2M
Chain-of-Custody and Shipping Forms	Verify the completeness of chain-of-custody records. Examine entries for consistency with the field logbook and sample processing log. Check that appropriate methods and sample preservation have been recorded. Verify that the required volume of sample has been collected and that sufficient sample volume is available for QC samples (for example, MS/MSD). Verify that all required signatures and dates are present. Check for transcription errors.	Internal/ External	TBD, Sampling Manager and Field Team Leader/CH2M TBD, Field Quality Manager, CH2M Shane Lowe, Project Chemist/ CH2M Laboratory TBD, QA officer
Analytical SOPs	Verify that the analytical SOPs were followed.	Internal/ External	Laboratory QA Officer/CLP Laboratory QA Officer/ASL Shane Lowe/CH2M
Laboratory Data	Verify that the laboratory deliverable contains all records specified in the QAPP. Check sample receipt records to ensure sample condition upon receipt was noted, and any missing/broken sample containers were noted and reported according to plan. Compare the data package with the chains of custody to verify that results were provided for all collected samples. Review the narrative to ensure all QC exceptions are described. Check for evidence that any required notifications were provided to project personnel as specified in the QAPP. Verify that necessary signatures and dates are present.	Internal	Shane Lowe, Project Chemist/ CH2M Herb Kelly, Sr. Chemist/ CH2M
Method QC Results	Verify that the required QC samples were run and met required limits.	Internal/ External	Laboratory QA Officer/CLP Laboratory QA Officer/ASL Shane Lowe Project Chemist/CH2M
Quantification Limits	Verify that the sample results met the project quantification limit specified in the QAPP.	Internal	Shane Lowe, Project Chemist/CH2M

Worksheet #36—Data Validation Procedures

The objective of the data validation is to assess the performance associated with the analysis in order to determine the quality of the data. Data validation will be accomplished by evaluating whether the collected data comply with the pre-defined project requirements (including method, procedural, or contractual requirements) and by comparing the collected data with criteria established based on the project DQOs.

All types of data, including screening data and definitive data, are relevant to the usability assessment. The following sections focus on the data review requirements for definitive data only.

Table 36-1. Validation Summary

Matrix	Analytical Group	Required Deliverable	Validation Percentage	Validation Criteria	Data Validator (title and organizational affiliation)
Soil	Metals	Level IV Data Report and EQuIS compatible EDD	100% Level III validation	Manual Stage 2A[a] and Stage 2B[a] validation per EPA National Functional Guidelines, laboratory SOPs, and QAPP criteria	ESAT/Shane Lowe, Project Chemist/CH2M
Soil	IVBA/Total Lead	Level IV Data Report and EQuIS compatible EDD	100% Level III validation	Manual Stage 2B[a] validation per EPA National Functional Guidelines, laboratory SOPs, and QAPP criteria	Shane Lowe, Project Chemist/CH2M

[a]Stage 2A and Stage 2B per, "Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use" (Office of Solid Waste and Emergency Response No. 9200.1-85, EPA 540-R-08-005, January 13, 2009)

36.1 Data Verification/Validation Scope Overview

CH2M will perform a Stage 2B data validation in accordance with the "Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use" (Office of Solid Waste and Emergency Response No. 9200.1-85, EPA 540-R-08-005, January 13, 2009), the site-specific QAPP, and laboratory SOPs on 100 percent of the laboratory-generated data from ASL and a Stage 2A validation for data generated (field QC only) from the CLP laboratory. ESAT will perform a Stage 2B data validation on 100 percent of the data generated from the CLP laboratory. EPA Functional Guidelines will be used as guidance for this data validation. No re-calculation of results will be performed.

CH2M will review the data validation reports against the data quality objectives to determine whether the data are acceptable. Additionally, a 10 percent comparison between the validated results and EDD will be performed to ensure comparability.

36.2 Field Data Review

Field-generated information may include field logbooks, boring logs, sample chain-of-custody forms, shipping documents, sampling observations, sample labels, Scribe exports, and other miscellaneous field observations. All field measurements and or field log information will be entered into field logbooks and reviewed daily by the field team leader or designee. The designee may be a qualified field geologist, engineer, environmental scientist, and/or technician.

36.3 Laboratory Data Review Requirements

All definitive data will be reviewed first by the laboratory analyst and then by the laboratory supervisor of the respective analytical section using the same criteria before they are submitted to CH2M. This internal data review process, which is multi-tiered, should include all aspects of data generation, reduction, and QC assessment. Elements for review or verification at each level must include, but are not limited to, the following:

- Sample receipt procedures and conditions
- Sample preparation
- Appropriate SOPs and analytical methodologies
- Accuracy and completeness of analytical results
- Correct interpretation of all raw data, including all manual integrations
- Appropriate application of QC samples and compliance with established control limits
- Documentation completeness (for example, all anomalies in the preparation and analysis have been identified, appropriate corrective actions have been taken and documented in the case narrative[s], associated data have been appropriately qualified, and anomaly forms are complete)
- Accuracy and completeness of data deliverables (PDF and electronic)

36.4 Laboratory Data Evaluation

The calibration, QC, corrective actions, and flagging requirements for definitive data are provided in Worksheet #28 (Analytical Quality Control and Corrective Action). Data qualifiers should be applied by the laboratory as part of their internal validation activities. Flagging criteria apply when acceptance criteria are not met and corrective actions were not successful or not performed. The data qualifiers must be reviewed by the supervisor of the respective analytical sections.

The laboratory QA section should perform a 100 percent review of 10 percent of the completed data packages. The laboratory project representative should complete a final review on all the completed data packages.

ESAT and the CH2M project chemist or designee will subsequently evaluate the flags applied by the laboratory as part of their data review and usability assessment activities. The flags may be accepted, modified, or rejected. For all data qualifiers that are changed, clear justification will be provided.

36.5 Data Verification Guidelines

The CH2M project chemist will review the data verification performed by the laboratory for completeness and accuracy. Data verification may be done electronically or manually, or by a combination of both. The verification process includes, but is not limited to the following:

- Sampling documentation (such as the chain-of-custody form)
- Preservation summary and technical holding times
- Presence of all analyses and analytes requested
- Use of the required sample preparation and analysis procedures
- The method detection and reporting limits will be evaluated against the project requirements
- The correctness of the concentration units
- Case narrative

36.6 Data Validation Guidelines

Data validation extends data verification and is used to confirm that the requirements for a specific intended use are fulfilled. Data validation is the systematic process of evaluating the compliance of the data with the pre-defined requirements of the project (including method, procedural, or contractual requirements) and compliance of the data against criteria based on the quality objectives documented in this document. The purpose of data validation is to assess the performance associated with the analysis in order to determine the quality of the data. Data validation includes a determination of the reasons for any failure to meet performance requirements, and an evaluation of the impact of such failures on the usability of the data. The project chemist may add or delete data qualifier flags during validation. Data validation guidelines have been developed in accordance with the method requirements, professional judgment and general EPA National Functional Guidelines requirements. The following information will be reviewed as part of a Level-III type summary data validation:

- Chain-of-custody documentation
- Holding time
- QC sample frequencies
- Method blanks
- LCS
- MS/MSD
- Initial and continuing calibration information
- FD precision
- Case narrative review and other method-specific criteria
- Laboratory duplicates
- Serial dilutions
- Post digestion spikes
- Calibration blanks

The verification/validation process will be performed by a combination of electronic and manual methods and includes data flagging for issues related to method blanks, laboratory control samples, field duplicates, surrogate recoveries, holding time, and reconciliation of dilutions and re-extractions. Data flags, as well as the reason for each flag, are entered into an electronic database and made available to the data users. A final flag is applied to the data by the data validator/chemist after evaluating all flags entered into the database and selecting the most conservative of the verification flags.

If, during the data review and verification process, a systematic problem or other major issue with the data is identified, the data validator/chemist will contact the laboratory's project manager or QA manager. Additional evaluation of the data may be performed including an in-depth review of the raw data to verify accuracy followed by analysis and interpretation of the data in the context of the project objectives and end-use as part of the usability assessment.

A data validation report will be prepared summarizing the findings and discussing their impact on the overall data usability. It will be incorporated into the final data evaluation summary report.

36.7 Flagging Conventions

The allowable final data qualifiers for definitive data and the hierarchy of data qualifiers, listed in order of the most severe through the least severe, are R, J, J+, J-, UJ, UB, and U. Their definitions are summarized in Table 36-2.

Table 36-2. Verification/Validation Data Qualifiers

Qualifier	Description
R	The data are rejected because of deficiencies in meeting QC criteria and may not be used for decision making.
J	Estimated: The analyte was positively identified; the quantitation is an estimation because of discrepancies in meeting certain analyte-specific QC criteria.
J+	Estimated high: The analyte was positively identified; the quantitation is a high estimation because of discrepancies in meeting certain analyte-specific QC criteria.
J-	Estimated low: The analyte was positively identified; the quantitation is a low estimation because of discrepancies in meeting certain analyte-specific QC criteria.
UJ	The analyte was not detected; however, the result is estimated because of discrepancies in meeting certain analyte-specific QC criteria.
U	Undetected: The analyte was analyzed for, but not detected or is qualified as nondetect because of blank contamination.

Table 36-3 presents the specific guidelines for applying these data qualifiers and includes additional information that is not included in the EPA National Functional Guidelines, but can be used to help define additional general flagging criteria applied (in some cases based on professional judgment).

Table 36-3. Data Qualifying Conventions—General

QC Requirement	Criteria	Flag	Flag Applied To
Holding Time	Time exceeded for extraction or analysis	J for positive results; R or UJ for nondetects*	All analytes in the sample
Sample Preservation	Sample not preserved (if sample preservation was not done in the field but was performed at the laboratory upon sample receipt, no flagging is required)	J for positive results; R or UJ for nondetects*	Sample
Sample Integrity	Temperature out of control	J for positive results; R or UJ for nondetects*	Sample
ICAL	All analytes must be within method specified criteria	J for positive results; R or UJ for nondetects*	All associated samples in analysis batch
Second Source Check or CCV	All analytes must be within method specified criteria	High Bias: J+ for positive results, no flag for nondetects Low Bias: J- for positive results, UJ for nondetects R for all nondetects greater than twice the control criteria	All associated samples in analysis batch
Low-Level Calibration Check or Interference Check Sample	All analytes must be within 20% of expected value	High Bias: J+ positive results, no flag for nondetects Low Bias: J- positive results, UJ nondetects R for all nondetects greater than twice the control criteria	All associated samples in analysis batch
LCS	% R > upper control limit %R < lower control limit %R <10%	J+ for positive results, no flag for nondetects J- for positive results; UJ for nondetects J- for positive results; R for nondetects	The specific analyte(s) in all samples in the associated batch

Table 36-3. Data Qualifying Conventions—General

QC Requirement	Criteria		Flag	Flag Applied To
Blanks (Method, Field, Trip or Calibration)	Blank Result	Sample Result		All samples in preparation, field or analytical batch, whichever one applies
	< RL	<RL	Flag U and raise to RL	
		≥ RL	Use professional judgment	
		<RL	Flag U and raise to RL	
	≥ RL	≥ RL but < blank result	U at sample concentration	
		≥ RL and ≥ blank result	Use professional judgment	
Field duplicates	Both sample results greater than 5 times RL and RPD greater than 50% or One or both samples less than 5 times RL and a difference between results of ±4 times RL for soil		J for positive results, no flag for nondetects	The specific analyte(s) in the associated sample Note: No flagging is required for results less than the reporting limit
Laboratory Duplicates	Both sample results greater than 5 times RL and RPD greater than 20%		J for positive results, no flag for nondetects	The specific analyte(s) in the associated sample
Matrix Spike/Matrix Spike Duplicates	%R > upper control limit		J+ for positive results, no flag for nondetects	The specific analyte(s) in the parent sample
	%R < lower control limit		J- for positive results; UJ for nondetects	
	MS/MSD %R <10%		J for positive results; R for nondetects	
	MS/MSD RPD > 20%		J for positive results, no flag for nondetects	
	Sample concentration > 4 times the spike concentration		No flag required	
	Excessive dilution*		No flag required	
Serial Dilutions (applicable to sample concentrations >50X RL)	All analytes must be within 10% of expected value		J positive results, UJ nondetects	The specific analyte(s) in the parent sample
	Sample concentration < 50x MDL		No flag required	
Post Digestion Spike (applicable when MS fails)	Within 75-125%		>UCL: J+ for the positive results <LCL: J- for the positive results; UJ for the nondetects	The specific analyte(s) in the parent sample

* Based on analyte-specific review

%R = percent recovery

Worksheet #37—Data Usability Assessment

The data usability assessment is an evaluation based on the results of data verification and validation in the context of the overall project decisions or objectives. The assessment determines whether project execution and resulting data meet the project DQOs. Both the sampling and analytical activities must be considered, with the ultimate goal of assessing whether the final, qualified results support the decisions to be made with the data.

The following subsections summarize the processes to determine whether the collected data are of the right type, quality, and quantity to support the environmental decision making for the project, and describe how data quality issues will be addressed and how limitations of the use of the data will be handled.

37.1 Summary of Usability Assessment Processes

Data gaps may be present if (1) a sample is not collected, (2) a sample is not analyzed for the requested parameters, or (3) the data are determined to be unusable. The need for further investigation will be determined on a case-by-case basis, depending on whether data can be extrapolated from adjacent sample locations, and whether the data are needed based on the results from adjacent sample locations.

The CH2M project chemist and the laboratory will confirm that the collected data meet the LODs, LOQs, and laboratory QC limits specified in this document. During the data validation assessment, nonconformances will be documented, and data will be qualified accordingly. The CH2M project chemist will determine whether the data are usable based on the requirements specified in this document.

The data as qualified by the validator/ project chemist are considered useable, with the exception of rejected data. Estimated and/or biased results are considered usable. Outliers, if present, can be addressed on a case-by-case basis. There is no generic formula for determining whether a result is an outlier. Potential outliers will be referred to a statistician and/or senior consultant, who will determine which formulas are appropriate for classifying data points in a statistically appropriate and defendable manner.

37.2 Evaluation Procedures to Assess Project-specific Overall Measurement Error

Overall measurement error is normally associated with both sampling design and quality and quantitative measures performed in both the field and laboratory. In-depth assessment will be performed during the data review and validation processes to assess conformance with the field SOPs, laboratory SOPs, and objectives of this document. Qualifiers will be used to indicate overall usability of the data.

37.3 Personnel Responsible for Performing Usability Assessment

- Shane Lowe, Project Chemist, CH2M
- Rachel Grand, SM, CH2M
- Barrie Selcoe, subject matter expert, CH2M
- TBD, sampling manager, CH2M
- Zach Dolbeare, field team leader and field quality manager, CH2M

37.4 Usability Assessment Documentation

The results will be assembled and reported for an overall quality assessment in a data evaluation report, which will be provided as an appendix to the RA report. The data validation report will identify precision and accuracy

exceedances with respect to the laboratory performance for each batch of samples, as well as comparability of field duplicates. Discussion will cover PARCCS criteria as described in the following subsections.

Precision

Precision is the measurement of the variability associated with the sampling and analytical process. It is determined by analysis of duplicate field and/or laboratory samples and measures variability introduced by both the laboratory and field operations. Field duplicate and MS/MSD samples should be analyzed to assess field precision at a frequency as described in Worksheet #20 (Field QC Sample Summary). Laboratory precision is measured by the variability associated with duplicate (two) or replicate (more than two) analyses, such as laboratory duplicate and LCS/LCSD samples. Multiple LCS analyses over the duration of the project can also be used to evaluate the overall laboratory precision for the project. In this case, the comparison is not between a sample and a duplicate sample analyzed in the same batch, but between LCSs analyzed in multiple batches.

The required control limits for LCSD and MSD laboratory precision for each method, matrix, and analyte are provided in Worksheet #15 (Reference Limits and Evaluation). The required control limits for laboratory duplicates are described in the individual methods in Worksheet #28 (Analytical Quality Control and Corrective Action).
A control limit of ± 50 percent will be used for original and field duplicate concentrations greater than five times the RL for soil matrices. For duplicate sample results, the precision is evaluated using the RPD. For replicate results, the precision is measured using the relative standard deviation (RSD). The formula for the calculation of RPD is provided below.

If calculated from duplicate measurements:

$$RPD = \frac{(C_1 - C_2)\, x\, 100\%}{(C_1 + C_2)/2} \qquad (1)$$

Where:

RPD = relative percent difference
C_1 = larger of the two observed values
C_2 = smaller of the two observed values

If calculated from three or more replicates, use RSD rather than RPD:

$$RSD = (s/\bar{y})\, x\, 100\% \quad (2)$$

Where:

RSD = relative standard deviation
s = standard deviation
$\bar{y}$ = mean of replicate analyses

Standard deviation, s, is defined as follows:

$$S = \sqrt{\sum_{i=1}^{n} \frac{(yi - \bar{y})^2}{n-1}} \qquad (3)$$

Where:

S = standard deviation
yi = measured value of the i^{th} replicate
$\bar{y}$ = mean of replicate analyses
n = number of replicates

Accuracy

Accuracy reflects the total error associated with a measurement. A measurement is considered accurate when the reported value agrees with the true value or known concentration of the spike or standard within acceptable limits. Analytical accuracy is measured by comparing the percent recovery of analytes spiked into an LCS and/or MS/MSD to a control limit. For many methods of organic compound analysis, surrogate compound recoveries also are used to assess accuracy and method performance for each sample analyzed.

Both accuracy and precision are calculated for each analytical batch, and the associated sample results are interpreted by considering these specific measurements. The formula for calculation of accuracy is included below as *%R* from pure and sample matrices. Accuracy requirements are listed for each method, matrix, and analyte in Worksheet #15 (Project Action Limits and Laboratory Specific Detection/Quantitation Limits).

The formula for calculation of accuracy is included below as percent recovery (%R).

$$\%R = 100\% \; x \left[\frac{S - U}{C_{sa}} \right] \qquad (4)$$

Where:

%R = percent recovery
S = measured concentration in spiked aliquot
U = measured concentration in unspiked aliquot
C_{sa} = actual concentration of spike added

For situations where a standard reference material is used instead of or in addition to matrix spikes:

$$\%R = 100\% \; x \left[\frac{C_m}{C_{sm}} \right] \qquad (5)$$

Where:

%R = percent recovery
C_m = measured concentration of standard reference material
C_{sm} = actual concentration of standard reference material

Representativeness

Representativeness is a qualitative term that refers to the degree in which data accurately and precisely depicts the characteristics of a population, whether referring to the distribution of contaminant within a sample, a sample within a matrix, or the distribution of a contaminant at a site. Representativeness is determined by appropriate program design, with consideration of elements such as sampling locations. Objectives for representativeness are defined for each sampling and analysis task and are a function of the investigative objectives. Assessment of representativeness will be achieved through use of the standard field sampling and analytical procedures. Decisions regarding sample locations process and numbers and the statistical sampling design are documented in Worksheets #10 (Conceptual Site Model), #11 (Project/Data Quality Objectives), and #17 (Sampling Design and Rationale).

Completeness (Statistical)

Completeness is a measure of the amount of valid data obtained compared with the amount expected under correct, normal conditions. It is calculated for the aggregation of data for each analyte measured as a compound of concern for the project objectives. Valid data are data that are usable in the context of the project goals. Completeness is calculated and reported for each method, matrix, and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. For completeness requirements, valid results are all results not qualified with an

R-flag after a usability assessment has been performed. The goal for completeness, based on specific project goals, is 90 percent.

Defined as follows for all measurements:

$$\%C = 100\% \; x\left[\frac{V}{T}\right] \qquad (6)$$

Where:

%C = percent completeness
V = number of measurements judged valid
T = total number of measurements

Comparability

Comparability is a qualitative indicator of the confidence with which one data set can be compared to another data set. The objective for this QA/QC program is to produce data with the greatest possible degree of comparability. The number of matrices that are sampled and the range of field conditions encountered are considered in determining comparability. Comparability is achieved by using standard methods for sampling and analysis, reporting data in standard units, normalizing results to standard conditions, and using standard and comprehensive reporting formats. Complete field documentation using standardized data collection forms supports the assessment of comparability. Historical comparability can be achieved through consistent use of methods and documentation procedures throughout the project. Assessment of comparability is considered subjective and the results should be interpreted by experienced environmental professionals with a clear knowledge of the DQOs and project decisions.

Sensitivity

Sensitivity is the ability of an analytical method or instrument to discriminate between measurement responses representing different concentrations. This capability is established during the planning phase to meet project-specific objectives. It is important to be able to detect the target analytes at the levels of interest. Sensitivity requirements include the establishment of various limits such as calibration requirements, instrument MDLs, and RLs. The project QA/QC on method requirements has been established to be compliant with the EPA National Functional Guidelines. Project-specific RLs are established in Worksheet #15 based on PAL objectives.

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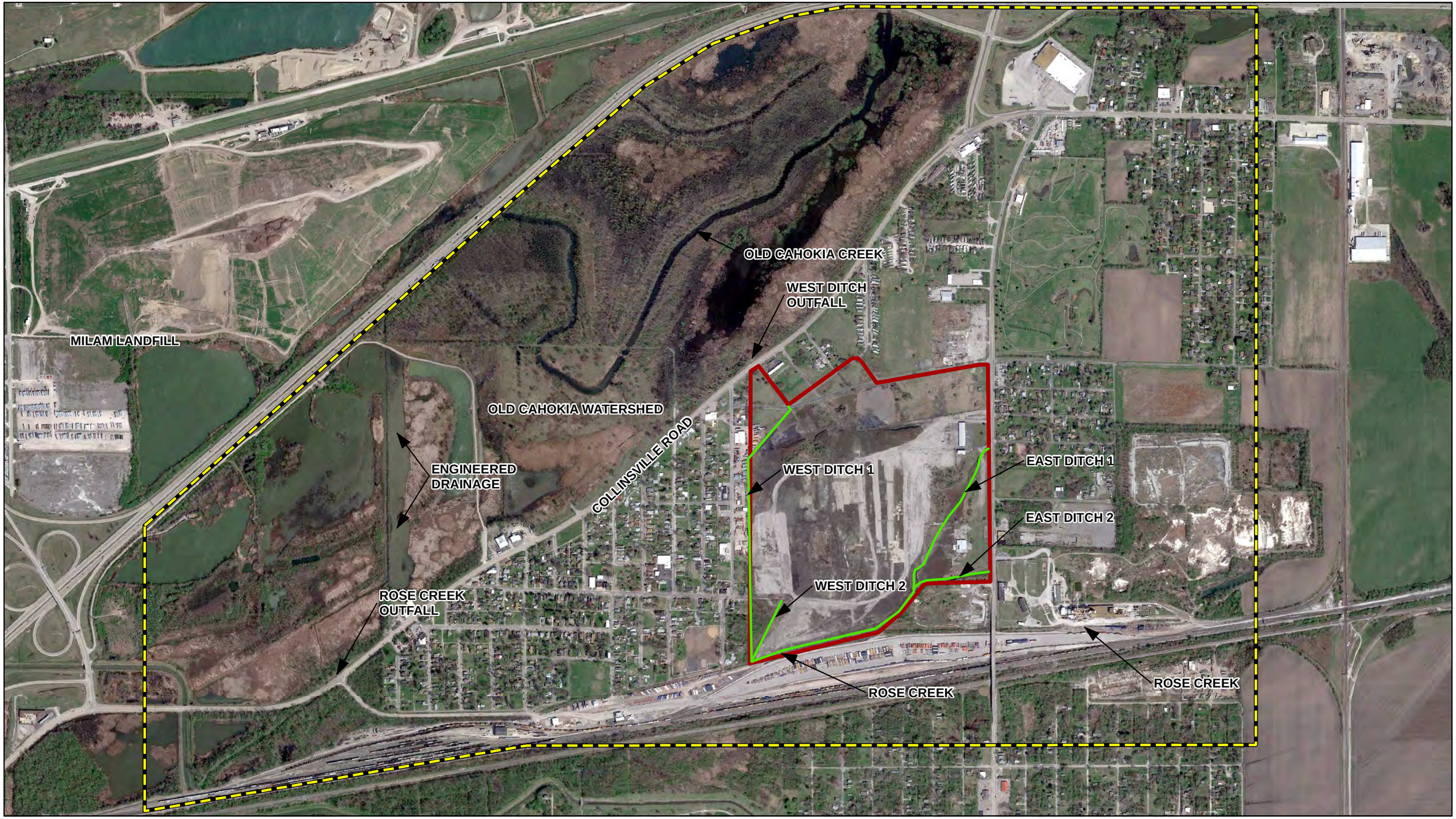
Figure



N

0 1,000 2,000

Feet

LEGEND

- EXISTING DITCH LINE
- FACILITY AREA BOUNDARY
- SITE PROPERTY (APPROXIMATE)

Notes:
1. Google earth Imagery Date: 4/2/2016.

DRAFT

Figure 1
Site Plan and Existing Conditions
Old American Zinc Site
Fairmont City, Illinois

ch2m

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Appendix A
Field Operating Procedures

Field Operating Procedure No. 1
Soil Sampling in Residential, Commercial and Vacant Areas

Old American Zinc Plant Superfund Site,
Fairmont City, St. Clair County, Illinois
Remedial Design
WA No. 224-RDRD-B5A1/Contract No. EP-S5-06-01

Revision Number: 0
Prepared: 3/10/2017

Approved By:

_____

Rachel Grand, Site Manager Date

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Theresa Rojas, Program Quality Manager Date

Sampling in Residential, Commercial or Vacant Areas

1.1 Purpose

This document describes the soil sampling process to be followed when sampling residential, commercial or vacant properties.

1.2 Scope and Applicability

This document describes the soil sampling process to be followed by CH2M HILL, Inc. when sampling residential, commercial, or vacant properties. The document discusses steps that must occur during sampling. This document should be reviewed by the field project team prior to working in the field.

Sampling will be performed using hand augers.

1.3 Equipment

- Hand auger
- Decontamination kits: distilled water, detergent solution, spray bottles, paper towels
- Laboratory-supplied bottleware
- Clean, unused garbage bags
- 5-gallon buckets
- Stainless steel spoons

1.4 Procedures

1.4.1 Properties with Less than 5,000-Square-Foot Surface Area

When sampling properties with a total surface area less than 5,000 square feet (a typical urban lot size), collect five-point composite samples from at least each of the following locations: the front yard, the back yard, and the side yard (if the size of the latter is substantial). Space the front, back, and side yard composites equally within the respective part of the yard and outside the drip zone, away from influences of other painted surfaces (Figures 1 and 2). Composites should consist of aliquots collected from the same depth interval.

From U.S. Environmental Protection Agency. 2003. *Lead-Contaminated Residential Sites Handbook.* August.

Figure 1. Recommended Minimum Soil Sampling in Yards Less Than or Equal to 5,000 Square Feet with Small Side Yard

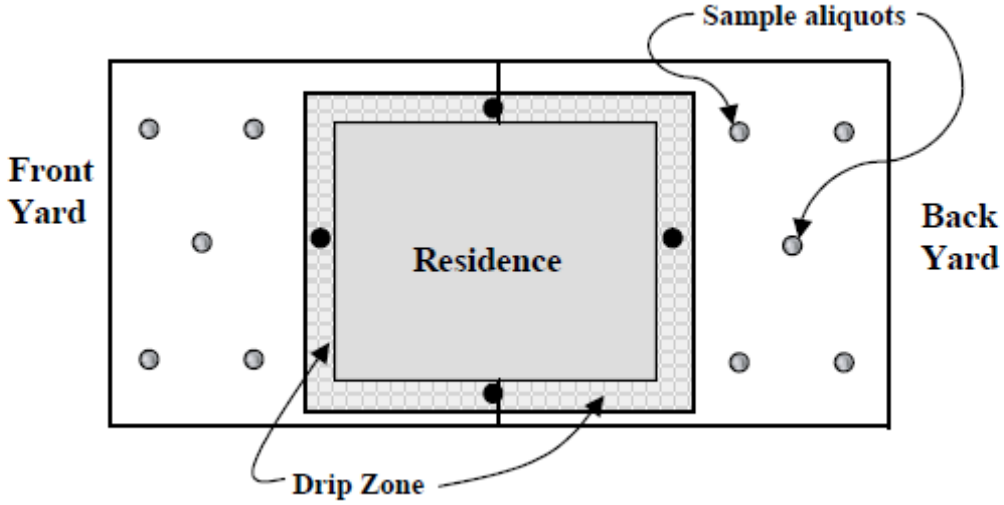
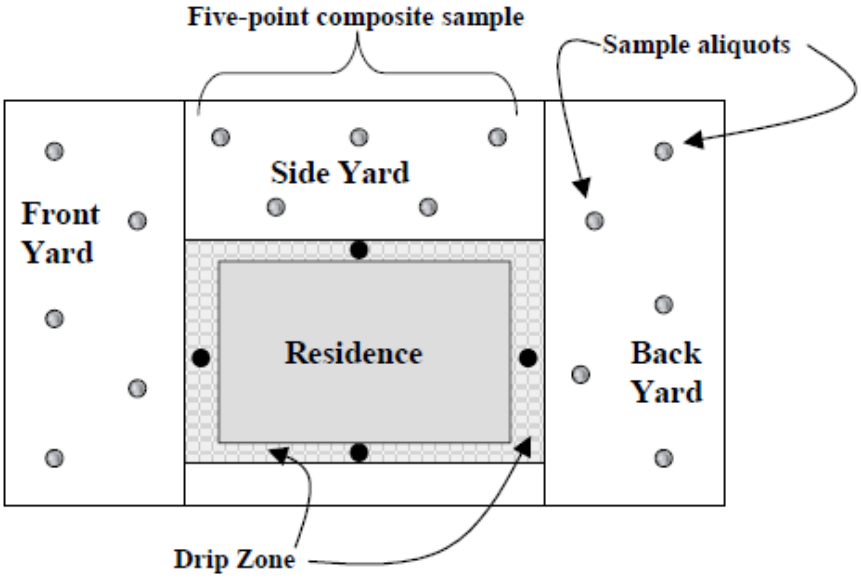


Figure 2. Recommended Minimum Soil Sampling in Yards Less Than or Equal to 5,000 Square Feet with Substantial Side Yard



Collect five-point composite samples from the front and back yards. Collect aliquots for a single composite sample from the same depth interval. Also, collect soil samples from distinct play areas and gardens if present, and from unpaved driveways and minimal use areas, such as areas under porches and crawlspaces. Space aliquot locations equally within the area of the yard where the composite is collected. Figure 1 illustrates one possible arrangement of the sample aliquots.

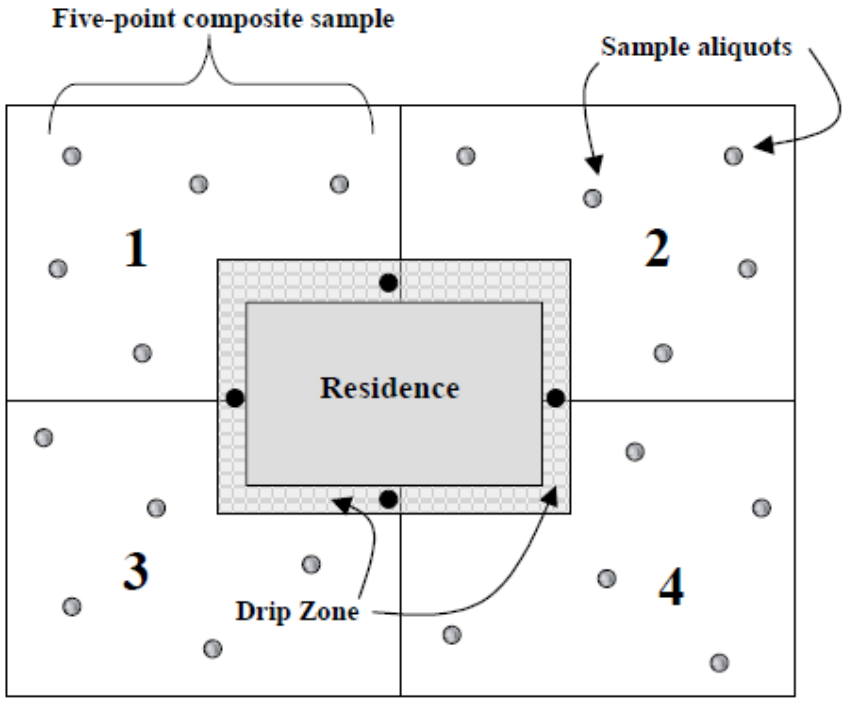
Collect five-point composite samples from the front, back, and side yards, along with other areas as described in Figure 1. Space aliquot locations equally within the area of the yard from which the composite is collected. The figure illustrates one possible arrangement of the sample aliquots. Collect aliquots for a single composite sample from the same depth interval.

1.4.2 Properties with Greater than 5,000-Square-Foot Surface Area

For lots with a surface area greater than 5,000 square feet, divide the property into four quadrants of roughly equal area. The two quadrants in the front yard should encompass one-half of the side yard, and so should the two quadrants in the back yard. One five-point composite of aliquots collected at equal spacing and from the

same depth interval should be obtained from each quadrant. Each aliquot should be collected away from influences of the drip zone and any other painted surfaces (Figure 3).

Figure 3. Recommended Minimum Soil Sampling in Yards Greater Than 5,000 Square Feet



Divide properties larger than 1 acre into 0.25-acre sections, and collect one five-point composite sample from each. For large properties, consider whether elevated concentrations trigger partial removal of soils or access restriction.

Collect five-point composite samples from each of the four quadrants as indicated above. Space the locations of the aliquots equally within each quadrant. Figure 3 illustrates one possible arrangement of the sample aliquots. The Drip Zone should not be sampled for the remedial design sampling event.

Composite samples should consist of discrete aliquots of equal amounts of soil. Collect the soil from each aliquot into a labeled garbage bag (used as a liner) in a 5-gallon bucket. Using a stainless-steel spoon, mix thoroughly. Once the sample is homogenized, place in the appropriate sample container. The sample can then be sent to the laboratory. Dispose of remaining sample volume in the general location from where it was collected, containerize, or archive, depending on the requirements of the project. In some cases, material other than grass and/or soil will be encountered at a sample location. For example, wood chips and sand often are found in recreational areas of day-care and school playgrounds. Samples of the soil below the cover material should be collected.

1.4.3 Sample Intervals

Sampling for select metals will be conducted to define the vertical extent of contamination for cleanup purposes at select properties. Composites should consist of aliquots collected from the same depth interval. Depth intervals include: 0 to 6 inches, 6 to 12 inches, 12 to 18 inches, and 18 to 24 inches below ground surface.

1.5 Quality Assurance/Quality Control

- Ensure samples are not collected within the Drip Zone.
- Ensure thorough mixing prior to analytical sample collection.

1.6 References

None.

Field Operating Procedure No. 2
Soil Sampling in Alleyways

Old American Zinc Plant Superfund Site,
Fairmont City, St. Clair County, Illinois
Remedial Design
WA No. 224-RDRD-B5A1/Contract No. EP-S5-06-01

Revision Number: 0
Prepared: 3/10/2017

Approved By:

_____

Rachel Grand, Site Manager Date

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Theresa Rojas, Program Quality Manager Date

Sampling in Alleyways

1.1 Purpose

This document describes the soil sampling process to be followed when sampling alleyways.

1.2 Scope and Applicability

This document describes the soil sampling process to be followed by CH2M HILL, Inc. when sampling alleyways. The document discusses steps that must occur during sampling. This document should be reviewed by the field project team prior to working in the field. In addition, this document should be used to develop scopes of work for drilling subcontractors.

Sampling will be performed using direct-push technology to drill through the asphalt or concrete surface.

1.3 Equipment

- Direct-push technology drilling rig
- MC-5 macrocores
- Decontamination kits: distilled water, detergent solution, spray bottles, and paper towels
- Laboratory-supplied bottleware
- Clean, unused garbage bags
- 5-gallon buckets
- Stainless-steel spoons

1.4 Procedures

1.4.1 Five-Point Sample Collection

When sampling alleyways, collect one 5-point composite per 2,500 square feet of alleyway (divide the alleyway in sections approximately 170 feet long, based on a 15-foot-wide alleyway). Space the 5 sample points equally in the alleyway sections, outside of any drip zones and away from influences of other painted surfaces. Composites should consist of aliquots collected from the same depth interval.

1.4.2 Sample Mixing

Composite samples should consist of discrete aliquots of equal amounts of soil. Collect the soil from each aliquot into a labeled garbage bag (used as a liner) in a 5-gallon bucket. Using a stainless-steel spoon, mix thoroughly. Once the sample is homogenized, place in the appropriate sample container. The sample can then be sent to the laboratory. Dispose of remaining sample volume in the general location from where it was collected, containerize, or archive, depending on the requirements of the project.

1.4.3 Sample Intervals

Sampling for select metals will be conducted to define the vertical extent of contamination for cleanup purposes at select alleyways. Composites should consist of aliquots collected from the same depth interval. Depth intervals include: 0 to 6 inches, 6 to 12 inches, 12 to 18 inches, and 18 to 24 inches below ground surface.

1.5 Quality Assurance/Quality Control

- Ensure samples are collected at the sample depth.
- Ensure thorough mixing prior to analytical sample collection.
- Ensure excess sample disposal in accordance with project requirements.

1.6 References

None.

Field Operating Procedure No. 3
In Vitro Bioaccessibility Sampling in Residential Areas

Old American Zinc Plant Superfund Site,
Fairmont City, St. Clair County, Illinois
Remedial Design
WA No. 224-RDRD-B5A1/Contract No. EP-S5-06-01

Revision Number: 0
Prepared: 3/10/2017

Approved By:

_____

Rachel Grand, Site Manager Date

_____

Theresa Rojas, Program Quality Manager Date

In Vitro Bioaccessibility Sampling in Residential Areas

1.1 Purpose

This document describes the soil sampling process to be followed when sampling residential properties for the *In Vitro* Bioaccessibility Study.

1.2 Scope and Applicability

This document describes the soil sampling process to be followed by CH2M when sampling residential properties. The document discusses steps that must occur during sampling. This document should be reviewed by the field project team prior to working in the field.

Sampling will be performed using an incremental sampling tool.

1.3 Equipment

- Incremental sampling tool
- Decontamination kits: distilled water, detergent solution, spray bottles, and paper towels
- Laboratory-supplied bottleware
- Clean, unused garbage bags
- 5-gallon buckets
- Stainless-steel spoons

1.4 Procedures

1.4.1 Surface Soil Sampling

One 30-point composite from one yard area per property will be collected. Aliquots collected at equal spacing and from the same depth interval (0 to 1 inch) should be obtained from one yard area, preferably from the yard where there is the most evidence of children playing. Aliquots will be collected using a clean, decontaminated incremental sampling tool in order to collect the same amount of material from each aliquot. Each aliquot should be collected away from influences of the drip zone and any other painted surfaces. It should also be noted if there is evidence of the soils being amended with phosphate (*does it appear that the property has been treated with fertilizer?*). Samples should not be collected from garden areas to avoid phosphate from fertilizers that affect the reliability of the analytical results.

1.4.2 Sample Mixing

Composite samples should consist of discrete aliquots of equal amounts of soil. Collect the soil from each aliquot into one clean container, such as a stainless-steel bowl, and mix thoroughly. Larger debris, organic litter, and sod should be removed from the composite sample. Once the sample is homogenized, place in the appropriate sample container. The sample can then be sent to the laboratory. Dispose of remaining sample volume in the

From U.S. Environmental Protection Agency. 2003. *Lead-Contaminated Residential Sites Handbook.* August.

general location from where it was collected, containerize, or archive, depending on the requirements of the project.

1.4.3 Sample Interval

Samples will be collected from 0 to 1 inches of soil below the organic litter and sod. Composites should consist of aliquots collected from the same depth interval.

1.5 Quality Assurance/Quality Control

- Ensure samples are not collected in drip zones.
- Ensure samples are not collected from garden areas.
- Ensure samples are not collected from properties amended with phosphate.

1.6 Attachments

U.S. Environmental Protection Agency. 2015. *Guidance for Sample Collection for In Vitro Bioaccessibility Assay for Lead (Pb) in Soil.* March.

1.7 References

U.S. Environmental Protection Agency. 2015. *Guidance for Sample Collection for In Vitro Bioaccessibility Assay for Lead (Pb) in Soil.* March.

United States
Environmental
Protection Agency

OSWER 9200.3-100

Guidance for Sample Collection for *In Vitro* Bioaccessibility Assay for Lead (Pb) in Soil

March 2015

Table of Contents

Guidance for Sample Collection for *In Vitro* Bioaccessibility Assay for Lead (Pb) in Soil

1.0 Terminology and Application

Definitions:

Absolute Bioavailability (ABA): Fraction of an ingested dose of lead that is absorbed from the gastrointestinal tract and enters the blood and tissues.

Relative Bioavailability (RBA): Ratio of the absolute bioavailability of lead in soil to that of a water soluble reference lead compound (lead acetate).

***In Vitro* Bioaccessibility (IVBA):** Fraction of total amount of lead in a soil sample that is soluble in a gastric-like (i.e., low pH) extraction medium.

The purpose of this document is to provide guidance on the collection of soil samples for measurement of lead IVBA (SW-846, Method 1340) (U.S. EPA, 2013c). The IVBA assay is used as a rapid and inexpensive method for predicting soil lead RBA (U.S. EPA, 2007c). Estimates of lead RBA are used to adjust bioavailability parameters in lead risk assessment models used in site risk assessment (e.g., Integrated Exposure Uptake Biokinetics [IEUBK] model for Lead in Children) (U.S. EPA, 2013a). Soil lead RBA is dependent on physical and chemical properties of the lead in soil and co-occurring elements at any particular site or location within a given site. As a result, site-specific estimates of soil lead RBA that provide representative coverage of the site are recommended for increasing confidence in estimates of risk related to site-specific lead exposures. Sampling plans for estimating soil lead RBA with the IVBA assay should provide a statistically robust estimate of RBA for decision units at the site. Typically, this can be achieved by measuring IVBA in a statistical subsample of soils collected as part of the sampling plan for estimating exposure point concentrations (EPCs) for soil lead. This guidance provides recommendations for data collection requirements, sampling material handling, QA/QC requirements, and health and safety requirements for assessments of site-specific soil lead RBA with the IVBA assay.

2.0 Procedure

Data Collection Requirements: A sampling plan for a site should be developed that considers potential soil exposure pathways for the site and any existing site data; for example, if the site is a residential area, then evaluation of exposure pathways in children's play areas, gardens, and the drip lines of homes should be given special attention (U.S. EPA, 2003a). If existing sampling data are available for a site, the information could assist in targeting the sampling locations where there is likely exposure to these contaminated areas.

Typically soil samples are collected, submitted for metals analysis, and the samples are archived while data are collected and reviewed. Based on the analytical results, a subset of the samples is selected for IVBA assay. At other sites, sample locations could be identified in the sampling plan and IVBA samples collected and analyzed without previous knowledge of lead concentrations at the site, although total metal analysis should be collected and conducted concurrently.

X-ray fluorescence (XRF) could be used to screen samples in the field because there is significant cost saving related to time and financial resources by eliminating the collection of samples that do not meet *a prior* criteria for IVBA analysis. There are many advantages of field screening for lead and other metals

including a reduction of both laboratory and field work. Soils with little to no metals are not collected, shipped, or processed by laboratory staff. Large fluctuations in soil lead concentrations within a site when determined by XRF in the field could be used as justification for collection of additional samples in order to form composites samples in the laboratory. The use of the XRF would allow samplers to immediately collect additional samples which may not be possible following laboratory analyses. Field screening with XRF therefore reduces the turnaround time required to generate IVBA results and reduces the need for additional field deployments as well as generating much less waste (fewer sample reduces shipping cost, processing time, number of analyses, and analytical waste). Field operators of portable XRF instruments should ensure they are following appropriate protocols to obtain reliable results (SW-846, Method 6200, U.S. EPA, 2007b).

When collecting samples for *in vitro* bioaccessibility assay, it is important to note site and sample medium characteristics that may indicate differences in the bioavailability of the lead or indicate that interferences might be present. For example, the lead IVBA assay (SW-846, Method 1340) may not reliably predict RBA of lead in soils that have been amended with phosphate (Scheckel et al., 2013). If phosphate at a site is a concern, it would be worthwhile to analyze the samples for the phosphate concentration. When collecting soils from residential properties it may not be advisable to make composite soil samples from a garden (potentially fertilized with phosphorus) with the surrounding property. Likewise it may not be advisable to composite soil samples from the drip line of a home (possible source of lead contaminated paint) with the remainder of the property (potentially different lead source).

In addition to total metals analysis and IVBA assay, the samples might also be submitted for lead speciation analysis and animal bioavailability studies. Lead speciation analysis is meant to determine the exact chemical form(s), or species, of lead in a sample, as opposed to the total lead concentration. Speciation analysis may be informative in explaining variability in IVBA across the site, identifying sources of contamination of the soil, and assessing the potential mobility of lead in the soil. While IVBA assay is meant to be a faster and less expensive alternative to *in vivo* animal bioavailability studies, there may be cases (such as potential interference from soil amendment applications [e.g., phosphate], untested lead phases, etc.) when the animal study would be necessary. It is important to ensure that sufficient material is collected for each sample so that additional analyses could be performed. If additional analyses are determined to be necessary, such as lead speciation analysis or *in vivo* animal bioavailability studies, consultation with the Technical Review Workgroup (TRW) Lead Committee is recommended.

Prior to sampling, a determination must be made as to whether the soil is regulated or quarantined by the U.S. Department of Agriculture (USDA) Animal and Plant Health Inspection Service (APHIS)/Plant Protection and Quarantine (PPQ) (USDA, 2014). Take special care to segregate regulated or quarantined soil samples from the non-regulated or non-quarantined samples. To determine if the soils collected are regulated or quarantined contact the State Plant Health Director (http://www.aphis.usda.gov/wps/portal/aphis/ourfocus/planthealth?1dmy&urile=wcm%3apath%3a%2Faphis_content_library%2Fsa_our_focus%2Fsa_plant_health%2Fsa_program_overview%2Fct_sphd).

Number of Samples: The number of samples to collect and analyze for IVBA will depend on the Data Quality Objectives (DQO) for the study. Factors that should be considered in estimating the number of samples include:

- goals of the RBA assessment;
- size and characteristics of the decision units at the site;
- expected variability in RBA within decision units, based on available data or bounding assumptions (U.S. EPA, 2007d); and
- acceptable limits on decision errors.

Project managers should consult with U.S. EPA "Guidance on Systematic Planning Using the Data Quality Objectives Process" or other appropriate guidance when developing DQOs (U.S. EPA, 2006).

In general, sample size estimates for RBA assessments can be based on the same types of power analyses used to evaluate statistical hypotheses in estimating EPCs at decision units (DUs) (see Appendix A). To reduce the cost of analyzing numerous discrete samples, an incremented sampling plan may be a cost effective approach (ITRC, 2012).

Sampling Depth: The appropriate sampling depth for a site will depend on the expected exposure pathway for a site. For most scenarios involving exposure to contaminated surface soil, EPA recommends a sampling depth of the top 0–1 inches of soil below organic litter and sod for lead exposure analysis (http://www.epa.gov/superfund/lead/ieubkfaq.htm). With this rather shallow sample depth it could be challenging to obtain sufficient sample mass for discrete samples especially if the material is particularly course. Incremental composite sampling can provide larger masses for shallow samples. If there are other exposure scenarios for a site, other sampling depth intervals that would represent these scenarios should be collected.

Sample Preparation: To help ensure that sufficient sample material is available for analysis, the field samplers should consider sieving the material in the field to remove larger debris. Sieve screens No. 4 (4.72 mm) or No. 10 (2.0 mm) would be sufficient for removing larger debris in the field.

Sample Mass: For metals analysis, SW-846 recommends that a minimum of 200 g of soil be collected and 2 g of sample be used for the digestions (SW-846, Chapter 3 Inorganic Analytes, Table 3-2, U.S. EPA, 2007a). Method 1340 specifies that 1 g of dried and sieved soil sample be used for IVBA assay for lead for a single replicate (U.S. EPA, 2013c). Additional replicates may be required if the assay does not meet performance specifications for IVBA. The amount of sample required will depend on the particle size distribution of the soil and the moisture content of the soil following course sieving in the field. If the samples will be submitted for animal bioavailability studies or speciation analysis, the laboratories that will be conducting these analyses should be consulted on the amount of sample materials they require to determine the sample mass needed. For further assistance in determining the sample mass for *in vivo* bioavailability and *in vitro* bioaccessibility assays, please contact the TRW Lead Committee.

Selection of Samples for IVBA: As stated previously, samples for IVBA assay can be designated as part of the sampling plan for estimating EPCs, or they can be selected based on the results from XRF field screening or total metals analysis. The strategy used to select samples for IVBA assay from XRF results or total metals data will depend on the intended use of the IVBA data. If the intended use is for screening, it may be appropriate to select only those samples that have lead concentrations exceeding the risk-based

concentrations used in screening. If the IVBA data are to be used to estimate risk for the site or a DU at the site, a representative statistical subsample should be selected. Samples selected for IVBA assay should have a total lead concentration less than 50,000 mg/kg (SW-846, Method 1340). If the *in vitro* bioaccessibility assay needs to be performed on a sample with a concentration greater than 50,000 mg/kg, the lab performing the assays should be informed of the samples concentrations so that the amount of soil used in the IVBA assay can be adjusted to be within the appropriate lead concentration range.

3.0 Sampling Materials and Handling

Sample Containers: The analytical laboratory/program that will be conducting the metals analysis should be consulted about the appropriate sample container and size required. For the *in vitro* bioaccessibility assay there are no specific sample container requirements. If no sample container is specified by the metals analysis laboratory, then appropriate containers include glass jars, wide-mouth HDPE jars, plastic zippered bags, or any other container that is clean and free of contaminants can be used. A single one-gallon plastic zippered bag should provide sufficient sample material for at least the metals analysis and *in vivo* bioaccessibility assay for most soils. Two-gallon plastic zippered bags may be required for sandy soils and soils with rocks passing through the sieve in the field. If using wide-mouth HDPE jars, a 1000-mL jar should provide sufficient sample, but collect multiple jars per sample if the soil is particularly course. There will be considerable cost reduction using plastic zippered bags compared to HDPE bottle (both cost of sample containers and shipping).

Sampling Equipment: Collection of surface soil samples may be accomplished with a stainless steel cylindrical punch which will capture a constant diameter core for the sampling depth of interest. Sampling using a kick-style cylindrical punch may reduce sample time in the field due to the ease of use. Kick-style punches are not recommended for sandy soils because the soil readily falls out of the probe. Likewise soils with heavy clay content or rocks are not recommended due to the difficulty in removing clay soils from punch and rocky soil will be rejected at the soil surface. For these reasons using plastic or stainless steel spades, trowels, or spoons may be preferable but the sampler should ensure that a sample is collected evenly across the sampling depth. Once the samples are collected, they should be placed in suitable containers for shipment. Any equipment that is not disposable should be thoroughly decontaminated and appropriately stored after sampling. If the exposure pathway being investigated requires deeper sampling depths than 0–1 inches, equipment such as augers, split spoon samplers, and backhoes may be necessary (U.S. EPA, 2000). If sampling at depth, care should be taken during sampling to account for any soil compaction as a result of sampling.

Labeling, Shipping and Storage Temperature, and Hold Times: Sample ID numbering, labeling, documentation, and chain of custody should follow the requirements of the analytical laboratory/program that will be conducting the metals analysis. The samples may be shipped at ambient temperature unless specified otherwise by the analytical laboratory/program.

EPA recommends a hold time of 6 months for metals samples. EPA 9200.2-86 recommends that all samples be archived after metal analysis and retained for further analysis, including *in vivo* bioavailability assay, for 6 months (U.S. EPA, 2012). The samples may be stored at ambient temperature unless specified otherwise by the analytical laboratory/program.

Laboratory Sample Preparation: Once in the lab, the samples should be blended and completely dried at <40°C in an air-drying oven for approximately 5 days to a constant mass. After drying, any clumps in the sample should be gently broken and then fine sieved. However, samples should not be ground by ball

mill or any other grinding method which could result in reduction in the particle sizes of the collected soils.

To ensure composite samples are representative of all of the component locations, the entire composite sample should be processed (i.e. dried and fine sieved). Following sieving, each sample should be thoroughly mixed using ASTM standard D6051-96 (2006) or ITRC Incremental Sampling Methodology (2012) and then transferred to a suitable storage container (U.S. EPA, 2013b).

Total metals analysis and other analyses should be conducted on the same dried, sieved, and homogenized sample material that will also be used for the *in vitro* bioaccessibility assay. To split a sample into equivalent aliquots for the different analyses, the processed soil should be passed through a riffle splitter and the aliquots collected in clean, 250 ml high-density polyethylene bottles (U.S. EPA, 2003b). Samples that have been dried and sieved can be submitted for total metals analysis, metals speciation, IVBA assay, and *in vivo* animal bioavailability studies but should not be used for analysis of other contaminants of concern.

4.0 Quality Assurance/Quality Control

The field samplers should consult with the metals analysis laboratory/EPA program to determine in advance the requirements for blanks, duplicates, and matrix spikes for the metals analysis samples. For the IVBA assay, Method 1340 does not require field blanks, field duplicates, or matrix spikes to be prepared or collected by field samplers. Material for the matrix spike and duplicates for Method 1340 can be taken from the samples at the laboratory's discretion and will not require that samplers collect and designate separate matrix spike and duplicates in the field.

Samplers should take thorough field notes and should retain any photographs taken, logbooks, and notes following the sampling event. The field group should make note of any differences in the media between the sample locations and indicate if there is any potential interferents (i.e., phosphate amended soils) present.

5.0 Health and Safety

When working with potentially hazardous materials, follow U.S. EPA, Occupational Safety and Health Administration (OSHA), and any contractor's corporate health and safety procedures, in addition to the procedures specified in the site-specific Health and Safety Plan.

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Appendix A

Guidance for Sample Collection for *In Vitro* Bioaccessibility Assay for Lead (Pb) in Soil

Objectives:

Predicting the minimum sample number needed to estimate the RBA-adjusted mean soil Pb concentration involves evaluating and setting limits on the probability of two types of errors. We define the null hypothesis as:

H_0: RBA-adjusted mean soil Pb concentration ≥ Risk based concentration (RBC)

And the alternate as:

H_1: RBA-adjusted mean soil Pb concentration < Risk based concentration (RBC)

A Type 1 error occurs if we reject H_0 when it is true; we conclude that the RBA-adjusted mean soil Pb concentration is less than the RBC, when it is actually greater than the RBC. A Type I error could result in underestimating risk at the site.

A Type 2 error occurs if we accept H_0 when it is false; we conclude that the RBA-adjusted mean soil Pb concentration exceeds the RBC, when it is actually less than the RBC. A Type 2 error could result in overestimating risk at the site.

The objective of a sample number assessment is to identify sample numbers that are expected to satisfy specified requirements for Type 1 and Type 2 error rates. These error rates depend on several factors:

- the difference between the mean soil Pb concentration and the RBC;
- the variability in the soil Pb concentration; and
- the mean and variability of the soil RBA at the site.

Larger sample numbers will be required to achieve a given error rate when the actual RBA-adjusted mean soil Pb is closer to the RBA, or when variability (i.e., standard deviation) of the soil Pb concentrations or RBA at the site are higher.

Assumptions for calculating sample number:

An example of sample number calculation is presented here. Assumptions in the analysis are as follows:

1. The underlying distribution of measured Pb concentrations in discrete soil samples at the decision unit (DU) is lognormal (the incremental-composite sampling [ICS] design should collect adequate samples to ensure a normal distribution of the concentrations of multiple composites).
2. Distribution of measured RBA within a DU is normal (e.g., single source of Pb contamination and uniform soil characteristics). An analysis of IVBA measurements of soil RBA at 10 different sites, at which multiple IVBA measurements were made (range: 12–86 discrete samples per site), showed that the average coefficient of variation (standard deviation/mean) was 13% (range: 5–22) (update to EPA TRW, 2003 that included data from Bunker Hill).
3. The RBA-adjusted mean soil Pb concentration for the DU is:

$$Adjusted\ Mean\ PbSoil = Mean\ PbSoil \cdot \frac{Mean\ RBA}{0.8}$$

where PbS is the soil Pb concentration and 0.8 is the default values for RBA in the IEUBK Model.

4. For evaluating Type 1 error, we assume that the RBA-adjusted mean soil Pb concentration at the DU exceeds the RBC. For evaluating Type 2 error, we assume that the RBA-adjusted mean soil Pb concentration at the DU is below the RBC.
5. An acceptable Type 1 error rate is 5% (i.e., the probability of concluding that the RBA-adjusted mean soil Pb concentration is less than the RBC, when it is actually greater than the RBC, is equal to or less than 5%).
6. An acceptable Type 2 error rate is 20% (i.e., the probability of concluding that the RBA-adjusted mean soil Pb concentration is greater than the RBC, when it is actually less than the RBC, is equal to or less than 20%). We are typically less concerned about a Type 2 error than a Type 1 error (overestimating risk) than a Type 1 (underestimating risk).
7. The ICS design consists of *n*=C composites are collected at the DU, each composite consisting of *n*=I increments, and *n*=R composites are randomly selected for IVBA analysis.
8. The estimated mean soil Pb concentration for the DU is the mean of measured Pb concentrations of *n*=C composites.
9. The estimated mean RBA for the DU is based on the mean of measured IVBA of *n*=R composites.
10. Values assumed for soil Pb concentration, RBC, and RBA for evaluating Type 1 and Type 2 error rates are presented in Table A-1.

A Monte Carlo Simulation (MCS) was used to estimate Type 1 and Type 2 error rates. The MCS consisted of 10,000 random draws from soil Pb concentration and RBA distributions (see Table A-1) and calculation of 10,000 corresponding values for the mean RBA-adjusted soil Pb concentration. The Type 1 error rate is the number of means that are less than the RBC (divided by 10,000) when the assumed (true) concentration equals or exceeds the RBC (see Figure A-1). The Type 2 error rate is the number of means that are greater than or equal to the RBC (divided by 10,000), when the true mean is less than the RBC.

Predictions:

Type 1 and Type 2 error rates for various ICS designs are presented in Table A-2. The single composite design is equivalent to a discrete sampling design with *I*=n discrete samples per DU. The estimated probability distribution of the RBA-adjusted mean soil Pb concentration for the sampling design *C*=3, *I*=20, and *R*=1 is shown in Figure A-1. A plot of the Type 1 error rates corresponding to various combinations of *C*, *I*, and *R* is shown in Figure A-2.

As noted previously, error rates depend on the values selected for the various parameters listed in Table A-1. This is illustrated in Figure A-3 which shows the probability of rejecting H_0 as a function of increasing mean RBA-adjusted soil Pb concentration for a design in which 3 composites of 30 increments each are collected. When the mean soil Pb concentration is well below 400 ppm (<200 ppm), the probability of rejecting H_0 is 100% (Type 1 error = 0). Similarly, when it is well above 400 ppm (>600 ppm) the probability of rejecting H_0 is 0% (Type 2 error = 0). However, at a soil Pb concentration of 500 ppm, the probability of rejecting H_0 is 5%, even though the mean exceeds the 400 ppm RBA (Type 1 error = 5%).

Figure A-3 also shows the effect of variability in RBA on the Type 1 error rate. Three coefficients of variation are shown (0.15, 0.30, 0.50). If the coefficient of variation is 0.50 (RBA=0.6±0.30), rather than 0.15 (RBA=0.6±0.09), the Type 1 error rate at a 500 ppm mean soil concentration increases from 5% to 18%. In order to decrease the Type 1 error rate to an acceptable 5%, the number of increments in each of the 3 composites would have to increase from 20 to 60. If the coefficient of variation is 0.30 (RBA=0.6±0.18), a 5% Type 1 error rate can be achieved with 25 increments in each of 3 composites.

Conclusions:

1. If the mean RBA-adjusted soil Pb concentration is 500±500 ppm and the mean soil Pb RBA is 0.60±0.09, an acceptable Type 1 error (5%) is predicted with:
    a. 1 composite made up of 60 increments;
    b. 2 composites made up of 30 increments; or
    c. 3 composites made up of 20 increments.
2. If 3 composites of 20 increments are collected, RBA assessment of a single randomly selected composite would yield an acceptable Type 1 error rate. A minimum of 30 increments has been recommended (ITRC, 2012).
3. Higher variability in RBA will require a larger number of increments per composite to achieve an acceptable Type 1 error rate.
    a. If the RBA coefficient of variation is 0.30 (RBA=0.60±0.18), 25 increments would be needed per composite.
    b. If the RBA coefficient of variation is 0.50 (RBA=0.60±0.0.30), 60 increments would be needed per composite.
4. A larger number of increments will be needed if the actual mean soil Pb concentration is closer to the RBC, and fewer will be needed if the actual mean Pb concentration is further from the RBC.
5. In general, for most risk assessment applications, acceptable Type I error rate can be expected if ITRC (2012) recommendations are followed (30 increments per composite).

TABLE A-1. Parameter Values for Sample Number Calculation

Parameter	False Negative Assessment	False Positive Assessment	Basis
Soil Pb RBC (ppm)	400	400	OSWER screening level corresponding to P_{10}=5% (approximately)
Mean RBA-adjusted soil Pb concentration (ppm)[a]	500	300	Assumption Type 1 error = RBC x 1.25 Assumption Type 2 error = RBC x 0.75
Mean RBC-adjusted soil Pb standard deviation (ppm)	500[b]	300[b]	CV=1 for Bunker Hill soil (or dust)
Mean soil RBA	0.60	0.60	Site-wide median (U.S. EPA OSRTI TRW)
Soil RBA standard deviation	0.09[c]	0.09[c]	CV=0.15, based on median CV for 11 sites (TRW: Estimation of Lead Bioavailability in Soil and Dust: Update to the Default Values for the Integrated Exposure Uptake Biokinetic Model for Lead in Children (11/02/11) plus Bunker Hill (CV=0.11)
IEUBK model default RBA	0.80	0.80	IEUBK Model

[a]RBA-adjusted mean soil Pb=Mean soil Pb x mean soil RBA/0.8, where 0.8 is the IEUBK Model default soil RBA
[b]Soil Pb distribution: lognormal (mean, SD).
[c]RBA distribution Normal (mean, SD, min, max), with min=0, max=1.

TABLE A-2. Error Rates for ICS Designs

Number of Composites for Pb Analysis (C)[a]	Number of Composites for IVBA Analysis (R)	Number of Increments per Composite (I)	Type 1 Error Rate (%)	Type 2 Error Rate (%)
1	1	10	29	14
1	1	20	18	8.2
1	1	40	9.0	3.1
1	1	50	6.6	2.0
1	1	60	4.1	1.3
1	1	80	3.2	0.5
2	2	10	18	8.1
2	2	20	8.5	3.3
2	2	30	4.5	1.3
2	2	40	2.6	0.5
3	3	5	22	10
3	3	10	13	4.8
3	3	20	4.6	1.3
3	3	30	1.8	0.5
3	1	5	23	10
3	1	10	13	5.4
3	1	20	5.3	1.5
3	1	30	2.3	0.6

[a]If we are interested only in estimating the mean soil Pb concentration (i.e., not the upper confidence limit of the mean), a single composite of *I*=n increments is equivalent to *I*=n discrete samples.

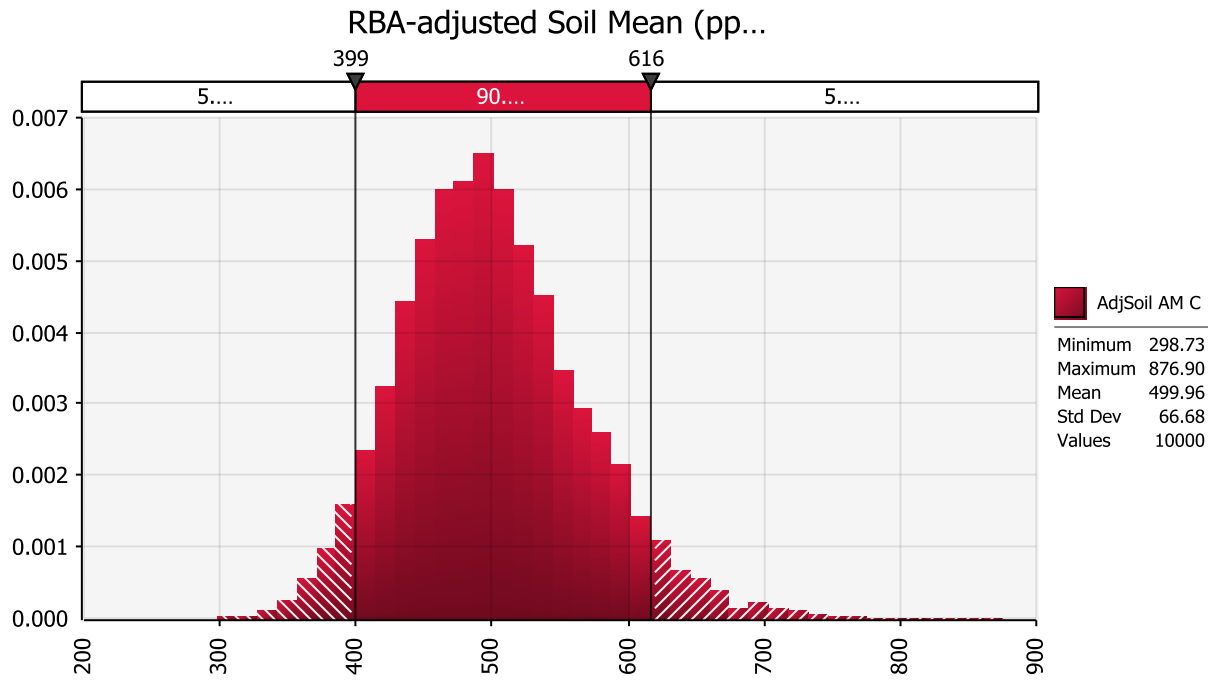


FIGURE A-1. Probability distribution (vertical axis) of estimated mean RBA-adjusted soil Pb concentration (horizontal axis) based on a 3 composite samples consisting of 20 increments with RBA measured on 1 randomly selected composite (C3xI20xR1). Cumulative distribution (percentile) is shown at the top of the graph. Actual soil Pb RBA is 0.60, actual mean soil Pb concentration is 500 ppm; RBC is 400 ppm. The probability of obtaining estimates that are less than 400 ppm (which would lead to Type 1 errors) is approximately 5%. In this case, a Type 2 error is not possible because the true mean exceeds the RBC.

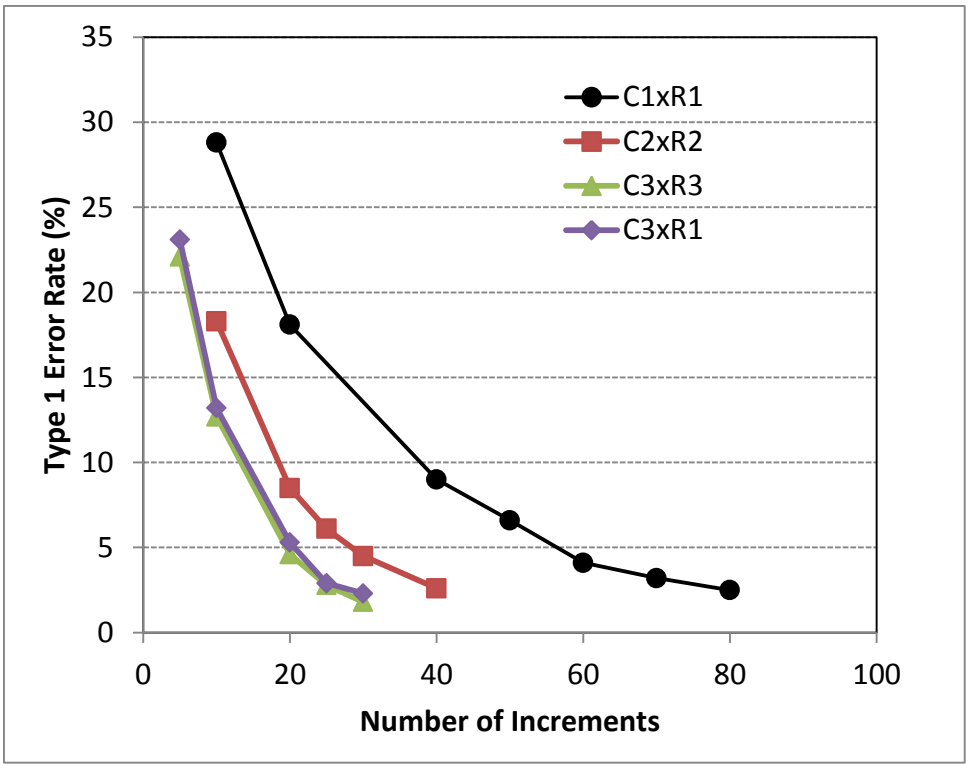


FIGURE A-2. Type 1 error rate (%) predicted for increasing number of increments for 1, 2, or 3 composite samples (20 increments per composite). Mean soil Pb RBA is 0.60±0.09, mean soil Pb concentration is 500±500 ppm; RBC is 400 ppm. The single composite design (C1xR1) is equivalent to a discrete sampling design with *I*=n discrete samples per DU.

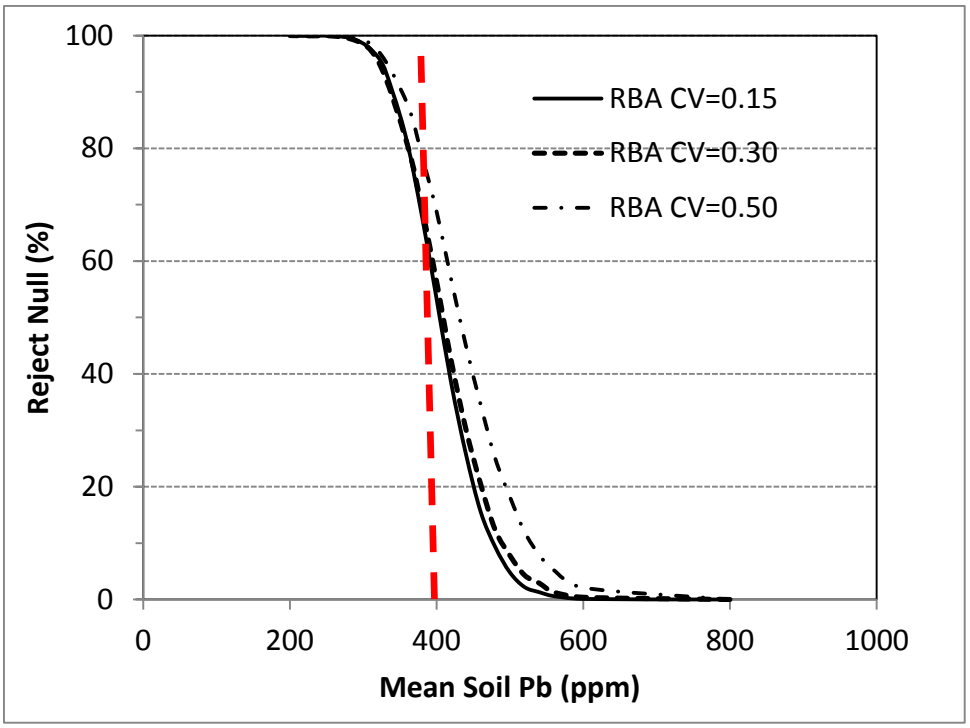


FIGURE A-3. Probability of rejecting H_0 as the mean RBA-adjusted soil Pb concentration increases when the coefficient of variation of RBA is 0.15 (RBA=0.60±0.09), 0.30 (RBA=0.60±0.18), or 0.50 (RBA=0.60±0.30). Soil Pb coefficient of variation is 1.0; RBC is 400 ppm. The area under the probability curve, to the right of the vertical line representing the RBC is the Type 1 error. Sample design is 3 composites of 20 increments per composite, with a single composite for RBA (C3xI20xR1).

Field Operating Procedure No. 4
Utility Clearance for Intrusive Operations

Old American Zinc Plant Superfund Site,
Fairmont City, St. Clair County, Illinois
Remedial Design
WA No. 224-RDRD-B5A1/Contract No. EP-S5-06-01

Revision Number: 0
Prepared: 3/10/2017

Approved By:

_____

Rachel Grand, Site Manager Date

_____

Theresa Rojas, Program Quality Manager Date

Utility Clearance for Intrusive Operations

1.1 Purpose

This document describes the utility markout/clearance process to be followed prior to completing any intrusive subsurface activities.

1.2 Scope and Applicability

This document describes the utility markout/clearance process to be followed by CH2M HILL, Inc. (CH2M) prior to any intrusive activities. The document discusses steps that must occur prior to mobilizing and steps that must occur during intrusive work.

This document should be reviewed by the field project team prior to working in the field. In addition, this document should be used to develop scopes of work for utility clearance subcontractors.

1.3 Procedures

1.3.1 Prior to Mobilizing to the Field

Prior to mobilizing to perform intrusive work, Illinois 811 (JULIE) must be called, and utility companies must mark their utility lines.

1.3.1.1 Public Utilities Under Illinois 811 (JULIE)

JULIE must be called (800.892.0123) prior to any intrusive work. JULIE members will be notified by JULIE and dispatch company representatives within 48 hours (2 business days) beginning at 8 a.m. and ending at 4 p.m. (exclusive of Saturdays, Sundays, and holidays recognized by the Statewide One-Call Notice System or the municipal one-call notice system). All requests for locates received after 4 p.m. will be processed as if received at 8 a.m. the next business day.

You may not begin your excavation before the dig start time of your ticket, even if all utilities have been marked. Excavation must begin within 14 calendar days of the initial locate request.

The following general information must be provided during the call:

- Your name, company, address, and phone and fax number where you can be reached.
- The name and phone number of the site contact.
- The county and city or county and unincorporated area of the township of the excavation.
- The location of the intrusive work, which may include, but is not limited to, address, cross street, lot numbers, etc. One shall provide either (1) appropriate section and quarter section grid information; (2) sufficient address or descriptive information to allow the establishment or drawing of a dig site polygon; (3) sufficient address, street, and cross-street information to allow for the determination of the appropriate section and quarter section grid(s); or (4) global positioning system coordinates for simple, single-site work areas for determination of a dig site polygon or appropriate section/quarter section during their locate request process through JULIE.
- The start date and time of the planned activities.

- The type and extent *(size of the excavation area)* of the work involved. Please indicate if the excavation area has been white-lined using white paint, flags, and/or stakes.
- The section/quarter sections when the above information does not allow the one-call system to determine this information.

While on the phone with JULIE, the following information should be collected and documented in the project files:

1. **Members Notified.** The identity of JULIE members notified will be provided to the caller. Retain a copy in the project files and keep it onsite while intrusive work is being performed.
2. **Case Reference Number.** An identification number associated with the call should be retained for future reference, if needed. Retain a copy in the project files and keep it onsite while intrusive work is being performed.

JULIE members are required to notify the contact person by fax or e-mail if they do not have any utilities at the work area. The site safety coordinator should retain a copy in the project records and keep it onsite while intrusive work is being performed.

All public utilities in the area where intrusive work is to be performed should be marked out on the ground by the utility locator using the American Public Works Association (APWA) Uniform Color Code (Attachment 1).

Utility locates are good for 14 calendar days, including the day the call was made. Extended tickets are available, and will be extended 14 calendar days from the extension. Extensions need to be requested 2 working days before the end of the original 14-day period. For more information, visit the website at www.illinois1call.com.

1.3.1.2 Private Utility Clearance

Private utilities must be cleared as follows:

1. The location(s) where intrusive work will occur should be identified during a site visit with the designated utility locator. The proposed areas where intrusive work will be performed should be pre-marked by CH2M before this site visit. It is important to consider access issues while pre-marking.
2. The utility locator should clear an area for intrusive work. An additional area (if possible) to be cleared by the utility locator is based on the work to be performed. When possible, the following areas will be cleared:
    - Boring: At some locations where utilities are congested, the boring location only will be cleared (i.e., only an “X” will be cleared). At other locations that are less congested with utilities, a 5-foot radius around the boring location may be cleared, allowing additional borings within that radius if refusal is encountered on the first attempt. This will be communicated on the utility clearance signoff sheet by the private utility locator. For borings that are located near certain lines (e.g., other water lines, etc.) and for which the property owner cannot pinpoint the line’s location, additional methods will be used to clear the location (see #4 under **Prior to Intrusive Work**).
3. The area that is cleared by the utility locator for intrusive work should include the marking of all utilities in the immediate area (at least a 10-foot buffer around the cleared area) using orange spray paint. If it is not possible to mark utilities, then a figure should be provided that will show the field team exactly where these utilities are located.
4. A utility clearance signoff sheet will be provided to CH2M after all areas, where intrusive work will be completed, have been cleared. The signoff sheet will document the location of all underground utilities and obstructions in the immediate area of the intrusive work (per #3, above). If the utilities could not be marked out on the ground, then a figure showing their location(s) should be provided with the signoff sheet.
5. The utility clearance is applicable for a 14-day period. Any intrusive work conducted after this 14-day period requires a new utility clearance.

1.3.2 Prior to Intrusive Work

The following should be completed before commencing intrusive work:

1. Verify that all public utility companies have identified the presence of utilities with marking paint or have provided a response back indicating the absence of utilities in the area. To verify what the utility markings on the ground indicate, use the color code in Attachment 1 (for public utilities). **If utilities have not been marked or a negative response has not been confirmed, do not perform intrusive work in that area. Sometimes utility companies do not have underground utilities near your dig area. In this case, check the JULIE Positive Response site at http://newtin.julie1call.com/newtinweb/ticketinfo.nas to ensure the utility company has cleared your dig site or responded to your call.**
2. Review the utility clearance signoff sheet to verify that the location has been cleared for intrusive work if private utilities are present. Also, review the markings on the ground and compare this against the document provided by the utility locator that indicates the utilities in the area. **If markings are missing or the signoff sheet or figure provided indicates that cleared areas contain utilities, do not perform any intrusive work until the utility locator has been contacted and has marked the missing utility line(s) or evaluated whether the cleared area contains utilities.**
3. Review the utility clearance documentation with the subcontractor during the tailgate meeting (if applicable).
4. Use other methods to identify utilities if there are numerous utility lines around the area and/or lines that cannot be clearly located where intrusive work is to be performed. If possible, hand digging or hand augering will be performed down to 4 feet below ground surface (bgs). Another method would involve the use of an air knife to bore 4 feet bgs with the use of high-pressure air that would not damage any utilities encountered.
5. Intrusive work can only be performed in the cleared area. If intrusive work needs to be performed outside of the cleared area, then the appropriate utility locator(s) must clear the new location. If the new area cleared involves private utilities, then an addendum to the initial utility clearance signoff sheet should be provided.
6. While performing intrusive work, monitor for signs of an encounter with a utility line. The signs include encountering fill material such as gravel, sand, or other fill material; warning tape; plastic; or metal. If, during the course of digging, a utility line has been exposed, it is your responsibility to inspect and support the utilities before backfilling. You must inspect utilities for any damage which could include the pulling or kinking of the utility or damage to the protective coating or covering. If damage exists, it is your responsibility to immediately notify the utility company directly. If there is any question about possible danger, we recommend contacting the utility company for instructions. Illinois rules only require you to contact Illinois 811 if you have reason to believe marks are incorrect or missing. You should also plan your work to minimize damage to markings.
7. If it is believed that a utility was struck, stop work and evacuate everyone if you have created a dangerous situation. If so, call 911 immediately and keep the area clear. Also, call the utility you hit and make them aware of what has happened. They can be reached by contacting JULIE at 800.892.0123
8. If refusal occurs while boring and it is believed **not** to be related to a utility, then advancement will be tried at another location within the cleared yard area.

1.3.3 What is my responsibility while I am digging?

After markings have been made, you are required to maintain a minimum horizontal (side to side) clearance of 2 feet (24 inches) between an unexposed utility and the cutting edge or point of any power operated excavating or earth-moving equipment. For example, if the markings indicate a 6-inch pipe is buried, the hand-dig zone is 54 inches wide (6 inches + 24 inches on each side of the mark). If excavation is required within the hand-dig zone, then the excavation must be performed very carefully, with hand tools, and without damage to the utility or undermining of lateral support. Please note that utility depths may vary due to installation practices, changes in

the grade, erosion, and other variables. Therefore, any depth readings given by a locator, if given at all, are only an indication of the approximate depth of the utilities.

1.4 Key Checks

Review checks outlined in the procedures above.

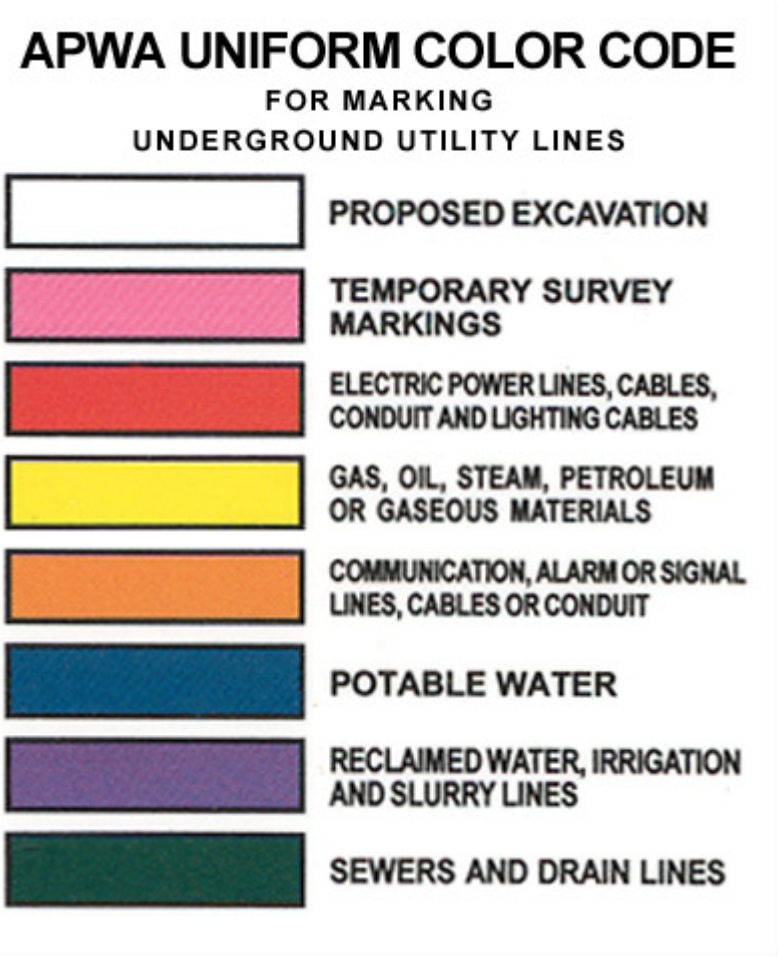
1.5 Attachments

APWA Uniform Color Code of Marking Underground Utility Lines

1.6 References

None.

Attachment 1



Field Operating Procedure No. 5 Sample Handling and Chain-of-Custody Procedure

Old American Zinc Plan Superfund Site,
Fairmont City, St. Clair County, Illinois
Remedial Design
WA No. 224-RDRD-B5A1/Contract No. EP-S5-06-01

Revision Number: 0
Prepared: 3/10/2017

Approved By:

_____

Rachel Grand, Site Manager Date

_____

Theresa Rojas, Program Quality Manager Date

FIELD OPERATING PROCEDURE FOP-05

Sample Handling and Chain-of-Custody Procedure

1.1 Purpose

The purpose of this field operating procedure (FOP) is to provide a definition of "custody" and describe protocols for documenting the transfer of custody from one party to the next (e.g., from the site to the laboratory). A documented custody trail is established through the use of sample tags and a U.S. Environmental Protection Agency (EPA) chain-of-custody form that uniquely identifies each sample container, and who has possession of it from the sample's origin to its final destination. The chain-of-custody form also describes the sampling point, date, time, and analysis parameters.

1.2 Scope

Sample personnel should be aware that a sample is considered to be in a person's custody if the sample meets the following conditions:

- It is in a person's actual possession.
- It is in view after being in a person's possession.
- It is locked up so that no one can tamper with it after having been in physical custody.

When samples leave the custody of the sampler, the cooler must be custody-sealed and possession must be documented.

Data generated from the use of this FOP may be used to support the following activities: site characterization, risk assessment, and evaluation of remedial alternatives.

1.3 Equipment and Materials

- Computer with Scribe software loaded
- Laser printer with paper (8.5 × 11 inch) and ink cartridge (black)
- EPA Region 5 Sample Tag
- Scribe generated tag label (2-inch x 4-inch adhesive labels)
- Indelible black ink pen

1.4 Procedures and Guidelines

For the Old American Zinc Plant Superfund Site Remedial Design, the following sample management tasks will be completed for each sampling task:

- **Preconstruction residential soil sampling:** Scribe will be populated by the sample manager. The sample manager will prepare chains of custody and tags.
- **Preliminary Data:** Preliminary electronic data deliverables will automatically be loaded into the EQuIS database and screened by the project chemist. The project chemist will send the data via email to the project team.

1.4.1 Chain-of-Custody Forms

The chain-of-custody form must contain the following information:

- CASE NUMBER/CLIENT NUMBER: If a Contract Laboratory Program (CLP) laboratory is used, then enter the case number provided by EPA's Regional Sample Control Coordinator (RSCC). If the CLP is not used, enter the Special Analytical Services (SAS) number provided by CH2M HILL Inc.'s (CH2M's) Sample and Analytical Coordinator.
- EPA REGION: Enter Region "5".
- CERCLIS ID: FOR OAZ, USE "IL0000034355".
- SPILL ID: For OAZ, use "TBD".
- SITE NAME/STATE: "OAZ", "IN".
- PROJECT LEADER: Enter the CH2M site manager.
- ACTION: For OAZ, choose "Remedial Design".
- SAMPLING CO.: "CH2M".
- SAMPLE NO.: This is the unique number that will be used for sample tracking. For CLP, this number is taken from a block of numbers assigned by the EPA RSCC. For non-CLP, the CH2M Sample and Analytical Coordinator will assign this number.
- MATRIX: Describes the sample media (e.g., soil, water, etc.).
- SAMPLER NAME: The name of the sampler or sample team leader.
- CONCENTRATION: Low (L), Low/Medium (M) or High (H).
- SAMPLE TYPE: "Grab" or "Composite".
- ANALYSIS: This indicates the analyses required for each sample.
- TAG NO.: This number appears on the bottom of the sample tag and includes a prefix ("5") followed by a series of numbers. The entire number must appear on the chain-of-custody form.
- PRESERVATIVE: Document what preservative has been added to the sample (e.g., "HCl", "Ice Only", "None").
- STATION LOCATION: This is the CH2M Station Location Identifier.
- SAMPLE COLLECT DATE/TIME: Use military time.
- QC TYPE: This is for field QC only, and includes field duplicate, field blanks, equipment blanks, and trip blanks.
- DATE SHIPPED: The date that samples are relinquished to the shipping carrier.
- CARRIER NAME: (e.g., "FedEx").
- AIRBILL: Airbill number used for shipping.
- SHIPPED TO: This is the laboratory name and full address, including the laboratory contact. If the contact is not known, use "Sample Custodian".
- CHAIN-OF-CUSTODY RECORD FIELDS: The sampler's signature must appear in the "Sampler Signature" and the "Relinquished By" fields. The date and time (military time) must also be included. If additional personnel were involved in sampling, their signatures should appear in the "Additional Sampler Signature(s)" field.

Although the samples are "relinquished" to the shipping carrier, the shipping carrier does not have access to the samples as long as the shipping cooler is custody sealed. Consequently, the shipping carrier does not sign the chain-of-custody form.

- SAMPLE(S) TO BE USED FOR LABORATORY QC: This identifies which samples are to be used for matrix spike/matrix spike duplicate analyses.
- INDICATE IF SHIPMENT FOR CASE IS COMPLETE: Use "Y" or "N".
- CHAIN-OF-CUSTODY SEAL NUMBER: Record the custody seal numbers that appear on the Region 5 custody seals that can be found on the shipping container. There is usually a minimum of two per shipping container.

1.4.2 Sample Tags

Each sample container will be identified with a uniquely numbered sample tag issued by EPA Region 5. Each tag will contain the following information:

- Case/SAS number
- The unique sample number for sample tracking
- CH2M station location (i.e., the sample identifier)
- Date of sampling
- Time the sample was collected (in military time)
- All parameters for which the sample will be analyzed
- Preservative used (if any)
- Sample type (grab or composite)
- Sample concentration (low, medium, high)
- Sample matrix (soil, water, etc.)
- The signature of sample team leader
- Identification when sample is intended to be used by the lab for matrix spike/matrix spike duplicate

1.5 Attachments

- Attachment 1: User Manual for Scribe CLP Sampling

1.6 Key Checks

- All sample containers must be properly tagged.
- Each cooler must have a chain-of-custody form and the samples in the cooler (as identified by the sample tags) must match what is on the chain-of-custody form.
- Verify completeness of the chain-of-custody form and consistency with field records.
- Each chain-of-custody form must be properly relinquished (signature, date, time).
- The custody seal numbers must be written on each chain-of-custody form.
- The shipping cooler must be custody sealed in at least two places.

1.7 References

None.

FOP-05, Attachment 1
User Manual for Scribe CLP Sampling

ERT

USER MANUAL for

SCRIBE CLP SAMPLING

TABLE OF CONTENTS

Modification Date: June 11, 2010

INTRODUCTION

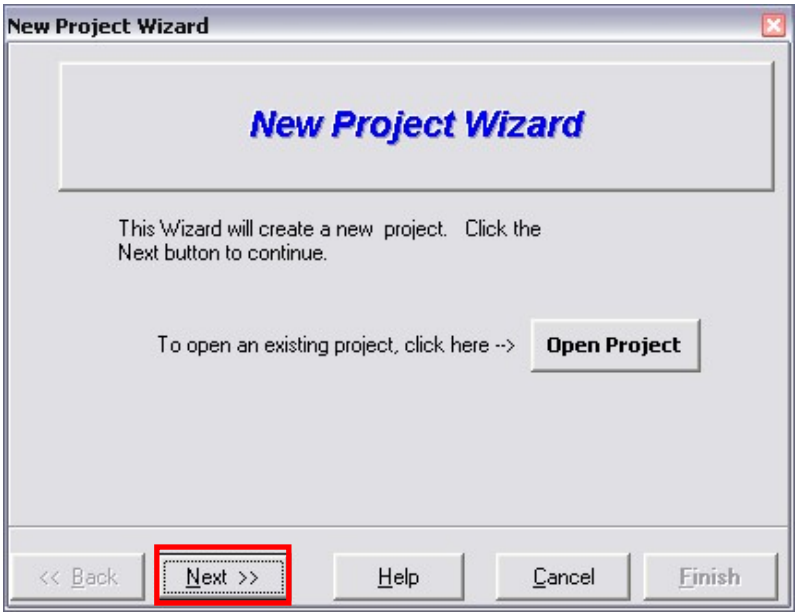
The intent of this User Guide is to provide a basic overview of how to use Scribe to create a new sampling project and manage samples collected for the EPA's Contract Lab Program (CLP). Scribe provides support for CLP sample documentation including the CLP Chain of Custody (COC) reports and the CLP XML format.Query. This document also assumes that the user is already familiar with the Scribe application for sampling. Otherwise, please refer to the Scribe User guides for detailed Scribe application instructions.

Create a New Project

New Project Wizard

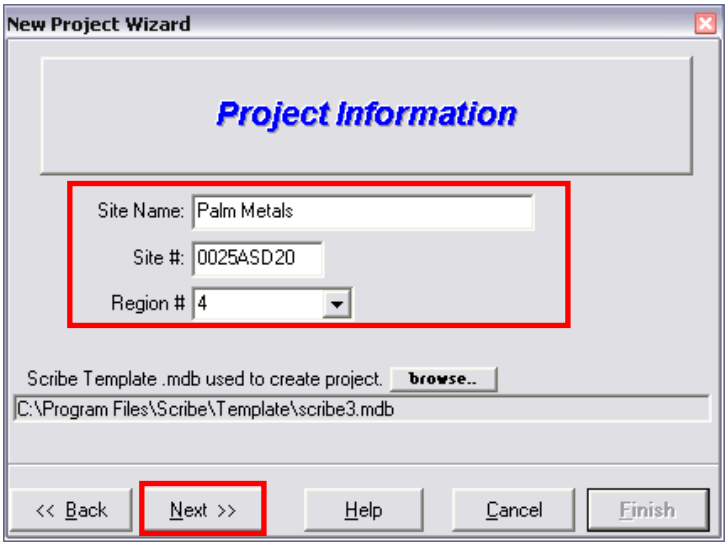
If you are starting Scribe for the first time after installation, the New Project Wizard will run automatically. Otherwise, to create a new project in Scribe:

1. Click on '**File'**.
2. Select '**New Project'**.
3. A New Project Wizard window is displayed.



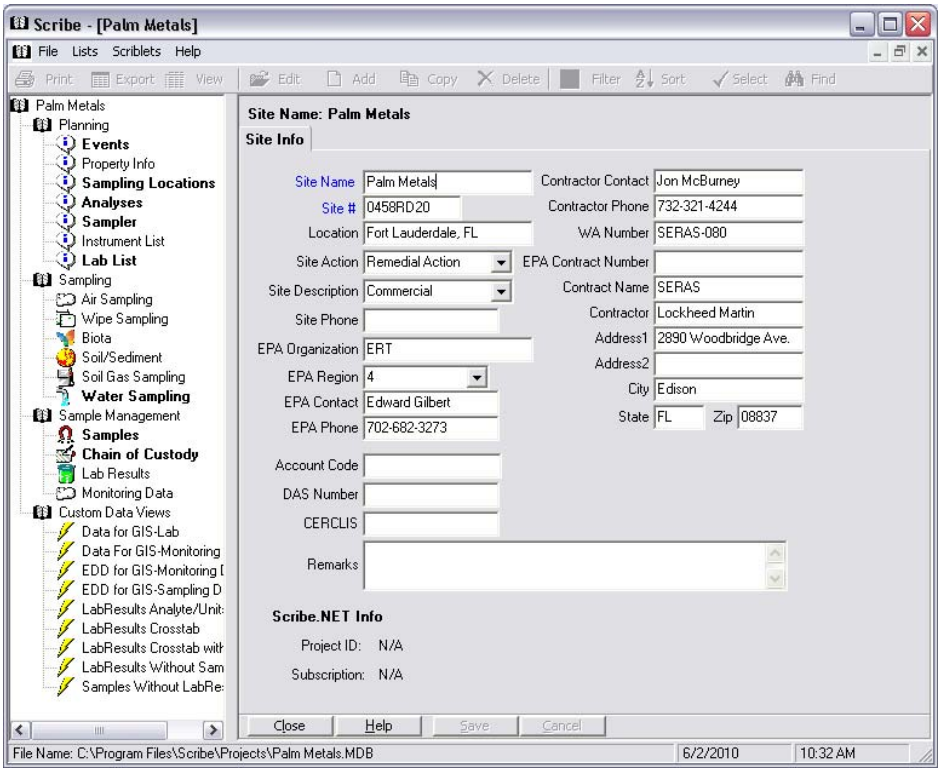
4. Click '**Next'** to continue.

5. Enter the Project Information.



6. Enter the **Site Name**, **Site #** and **EPA Region #**.

7. Click '**Next**' and then click '**Finish**' to create the new project.



The New Project Wizard closes and the "**Site Info**" screen displays. ONLY the field names in **BLUE** are required but we recommend completing as many fields as possible.

CLP SAMPLING IN SCRIBE

CLP Samples

CLP Analyses

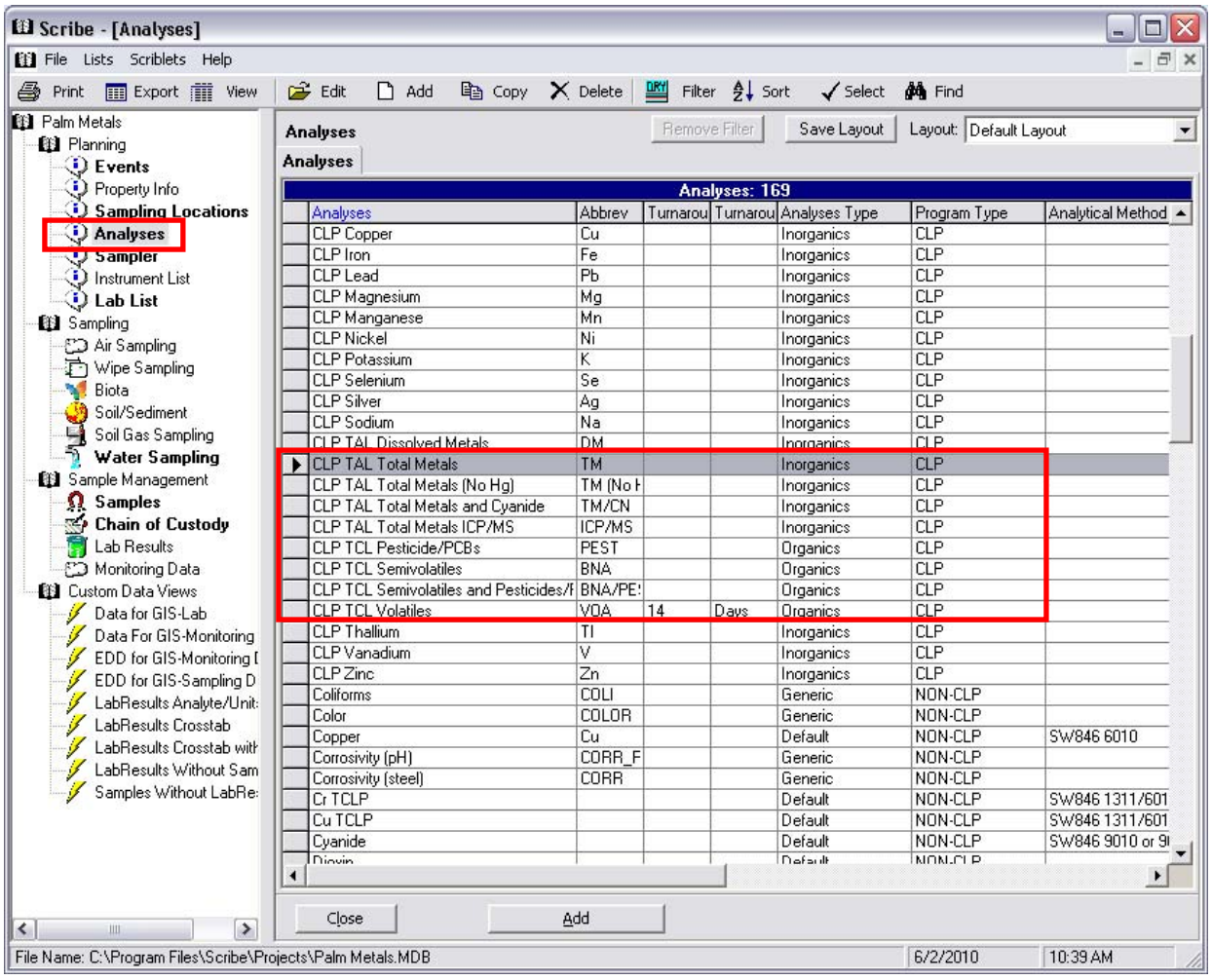
The Scribe Analyses List now includes CLP Analyses. To view or modify the list:

1. Click on "**Analyses**" in the left Navigation Pane. This section is used to manage a list of Analyses including the Program Type and Analysis Type. For example:

   Analysis: CLP TAL Total Metals

   Program Type: CLP

   Analyses Type: Inorganics



CLP/Tag Settings

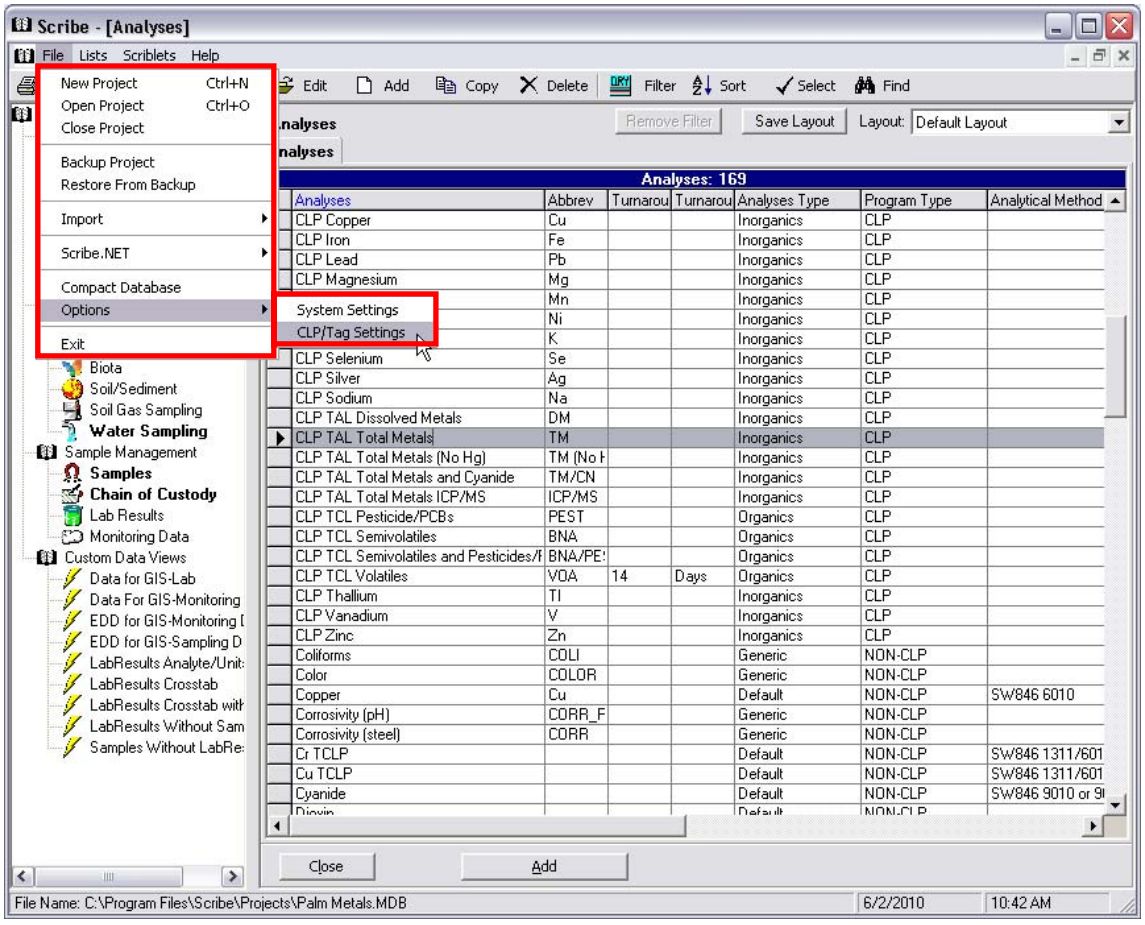
A new feature included with CLP Analyses is the ability to set defaults for the CLP Tags. When a CLP Analysis is selected for a sample, Scribe will assign a CLP Sample number. You can set the **Next CLP Sample number** and **Next Tag number** similar to a sample mask but not exactly.

The CLP Sample # and the Tag # is a field that will update as Samples are added to Scribe. This number is a DISPLAY of the Next number to be assigned. It is editable so that you may customize the next CLP Sample Number that you would like Scribe to assign to your samples.

The numbers auto-increment as samples are added using the CLP business rules.

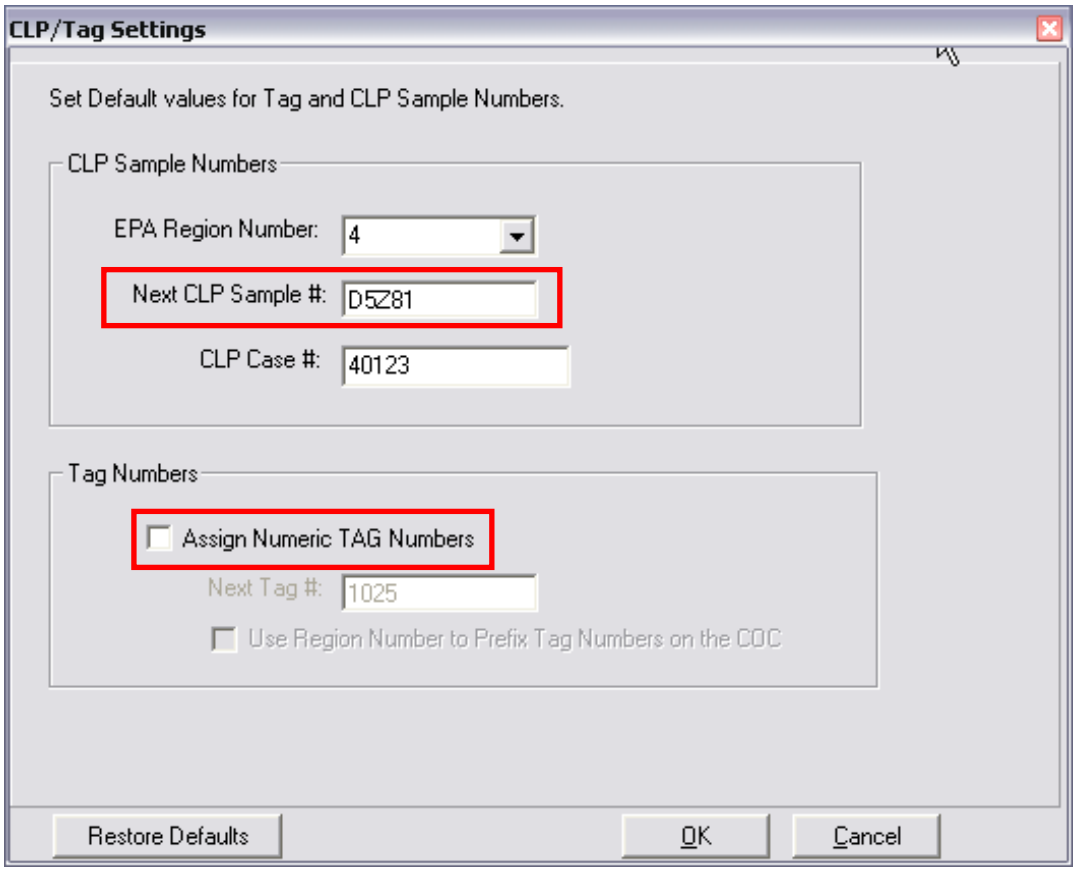
To modify the default settings:

1. Click on **File**.
2. Select **Options**.
3. Select **CLP/Tag Settings**.



4. The window for CLP/Tag Settings is displayed.

5. Input the appropriate information and click the '**OK**' button to Save and Close.



Adding CLP Samples and Assigning Analyses

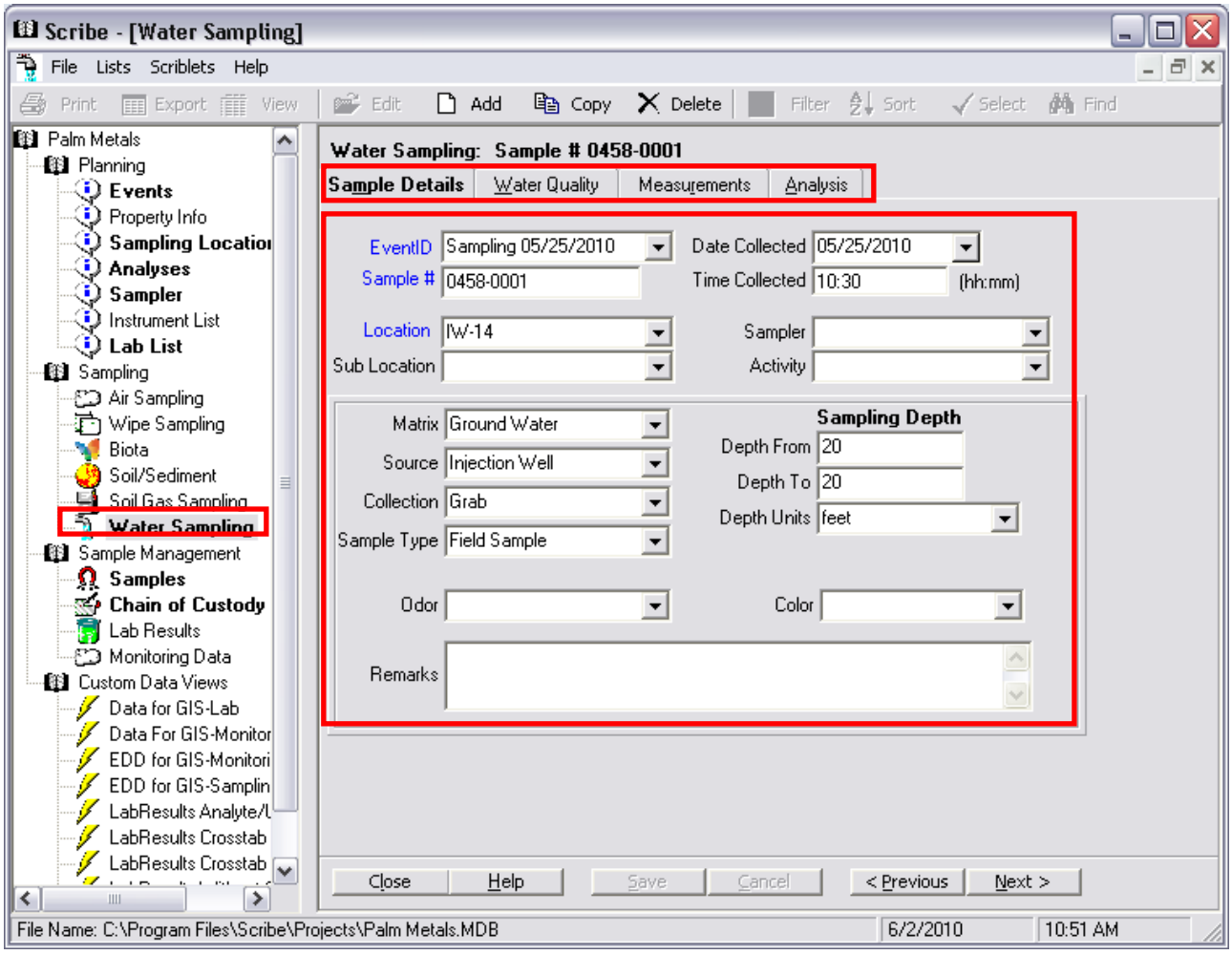
Depending on the type of sampling, click on the appropriate sampling task under Sampling in the left Navigation Pane. For example,

1. Click on '**Water Sampling'** in the left Navigation bar.

2. To add a Water Sample, click the '**Add**' button on the top menu.

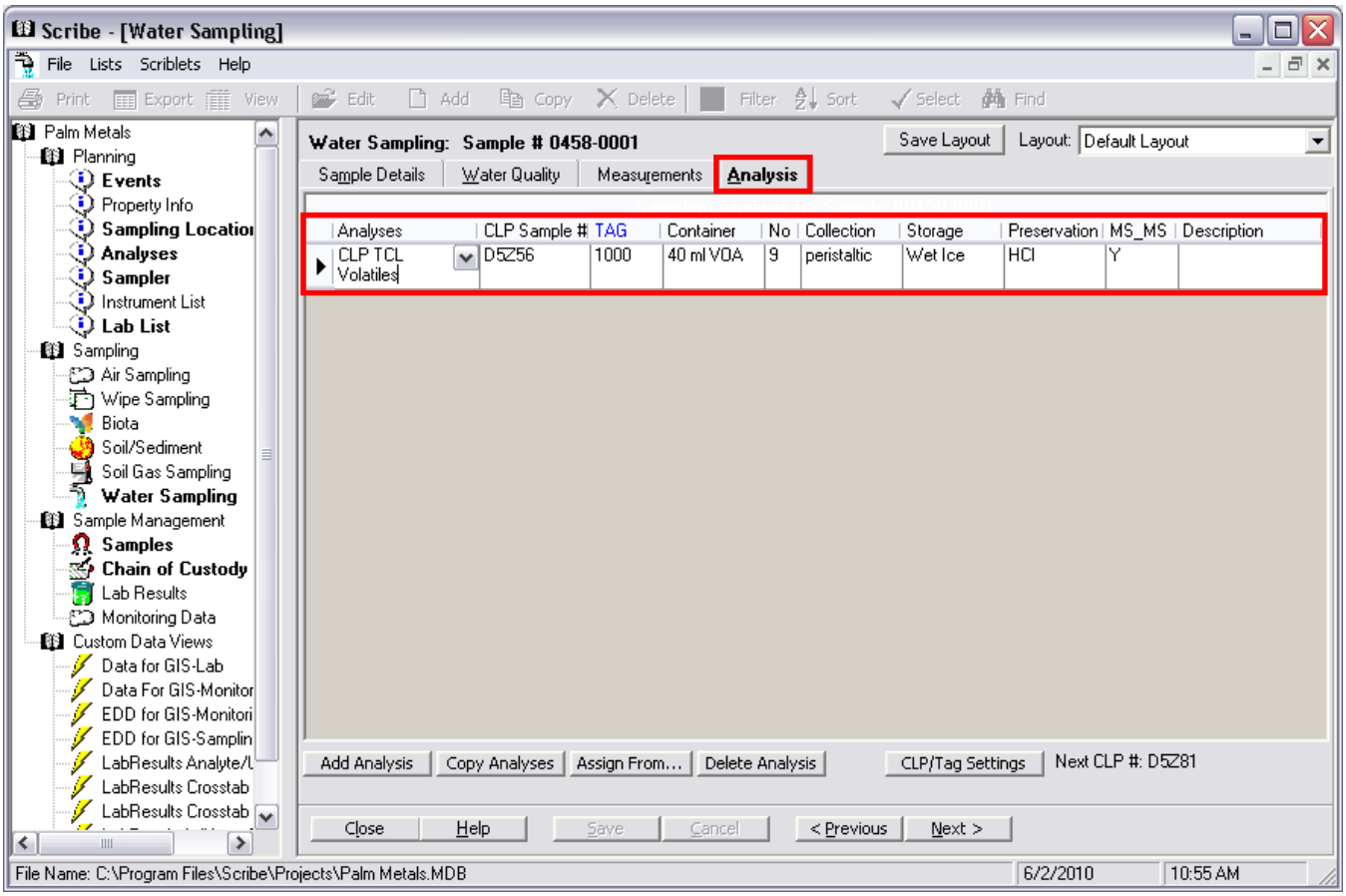
3. Enter sample information into the "**Sample Details"** screen.

Note: There are additional detail screens on the Water Quality and Measurements tabs. These tabs vary by sampling task. The details on the **Analysis** tab must be completed to assign an analysis to your sample.



Enter Analysis information for the Sample and assign CLP Sample and Tag numbers.

4. Click on the **Analysis** tab.
5. Click in the **Analyses field**.
6. Click on the **down arrow** for a list of the CLP Analyses that we referred to earlier.
7. Select an Analysis.



8. For a CLP Analysis, a Tag number and a CLP Sample number is assigned based on the CLP/Tag Settings.
9. To assign additional Analyses to sample containers, click the '**Add Analysis'** button.
10. When all analyses have been added, click the '**Close**' button on the bottom of the window to save and close.

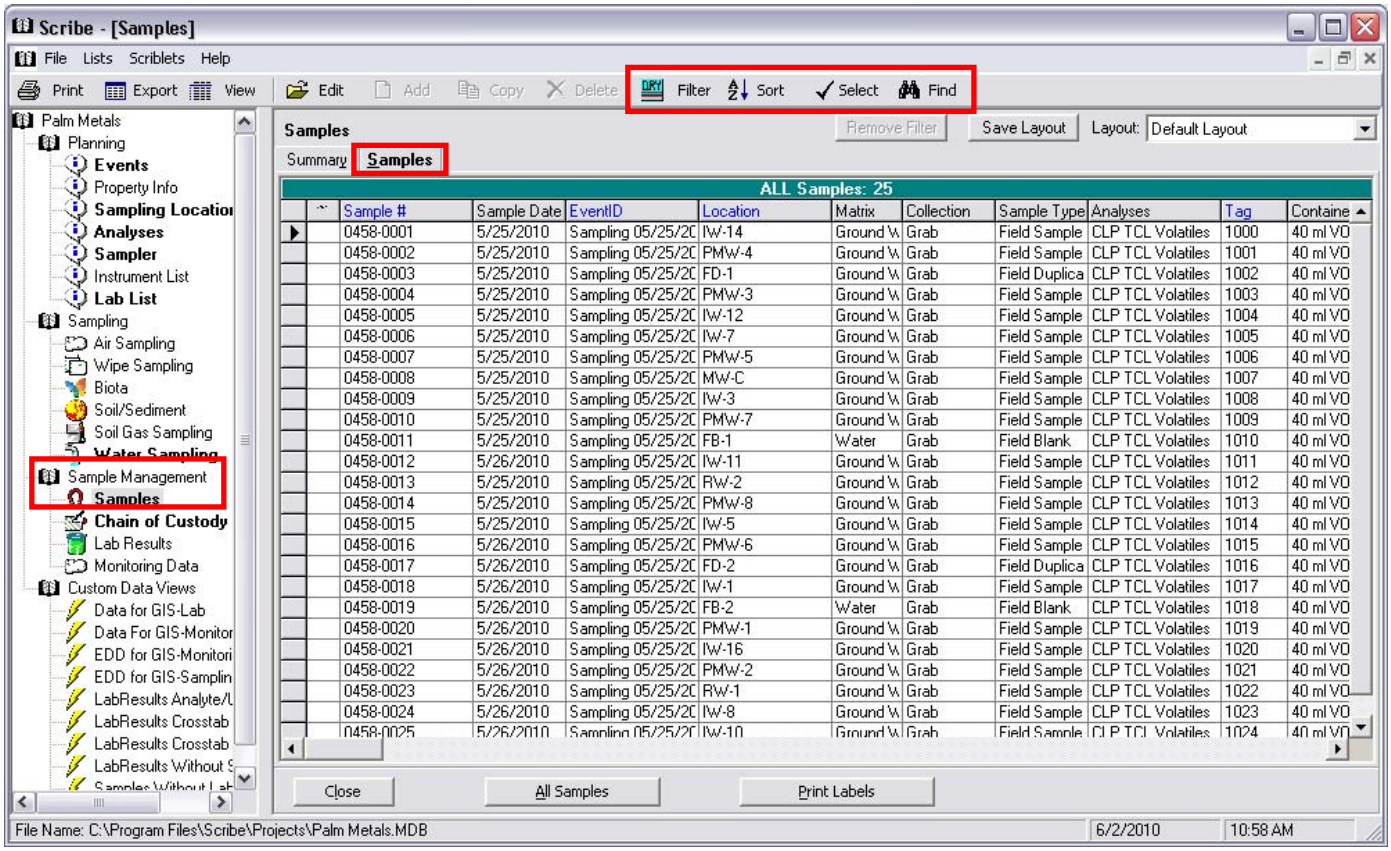
View Samples

Sample Management

Under Sample Management in the left Navigation Pane, you can view and manage all samples using Find, Filter and Sort. The options to Print labels and Chains of Custody are also available.

To view samples:

1. Click on '**Samples**' under Sample Management in the left Navigation Pane.

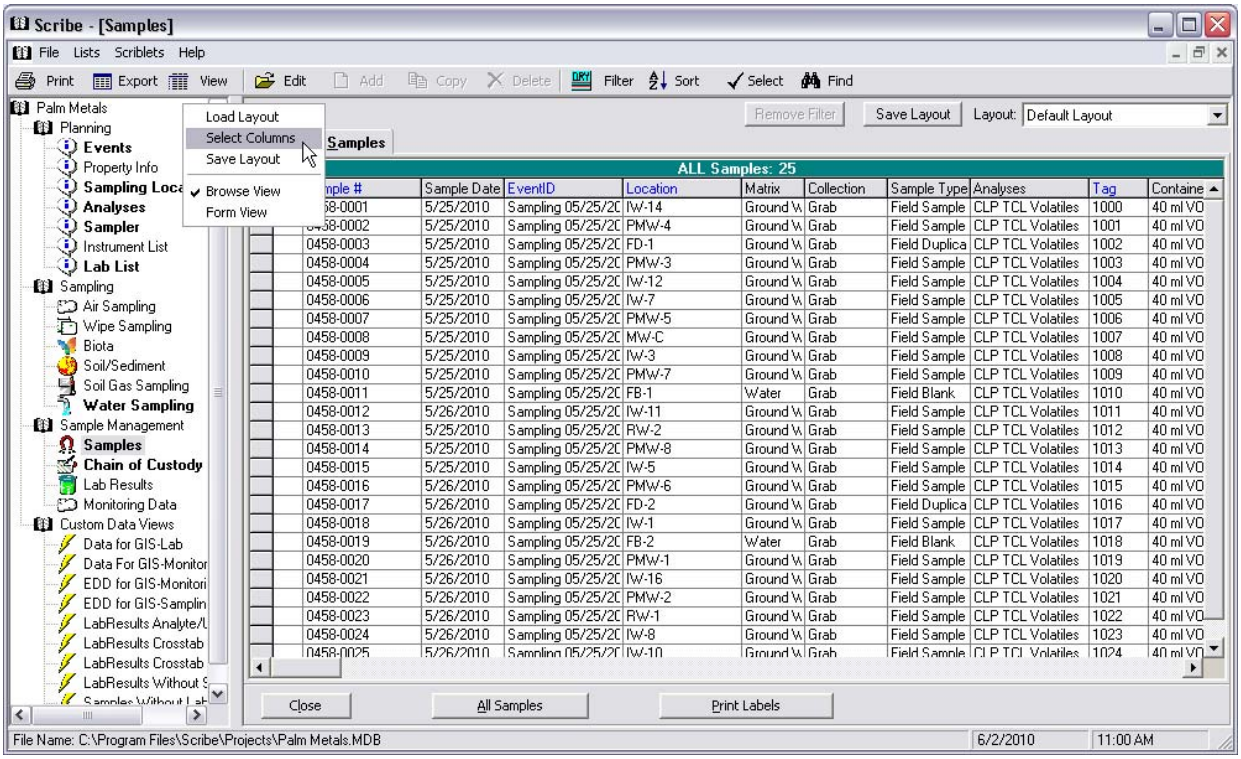


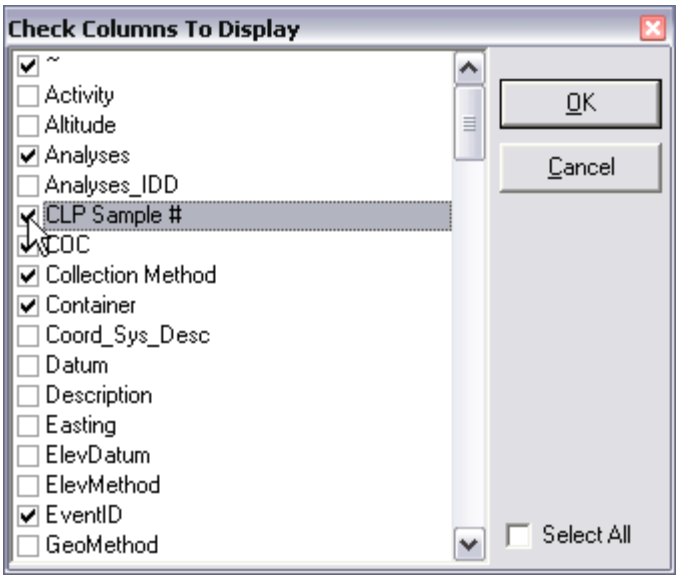
2. To filter your view of samples, RT-click on the field to filter on and select the '**Filter for…'** option. For multi-level filters, click the '**Filter'** button on the top menu bar.

3. To sort your view of samples, RT-click on the column heading and select a sort option. For advanced sort options, click on the '**Sort'** button on the top menu bar.

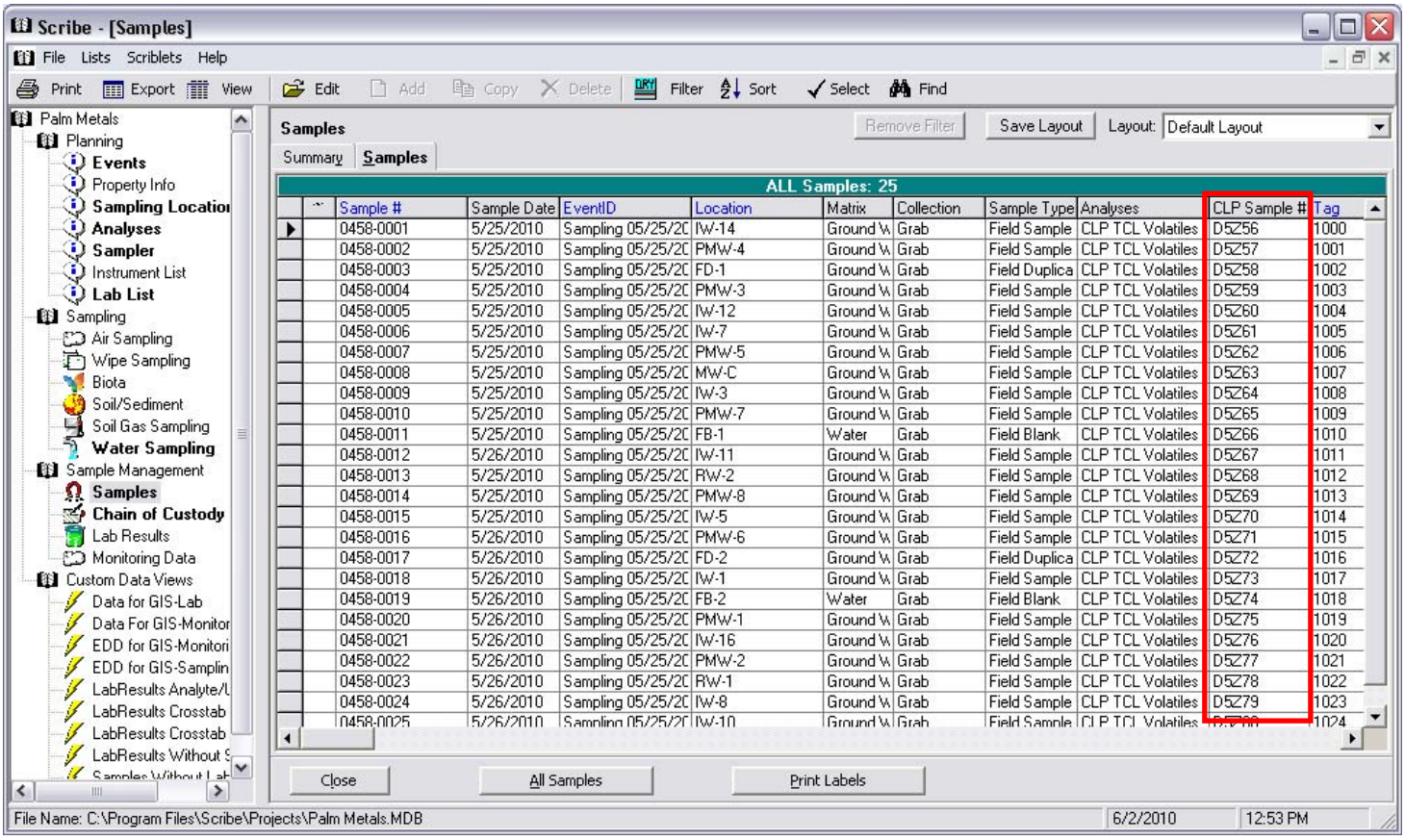
4. To find a particular sample(s), RT-click on the field and select the appropriate option. For multi-level finds, click the '**Find'** button on the top menu bar.

5. To see CLP Sample information including the **CLP Sample #,** click the drop-down menu for the Layout field on the top right corner of the window and select the '**CLP Layout**'.





6. The **CLP Sample #** column is now exposed.



LABELS AND CHAIN OF CUSTODY

CLP Sample Labels

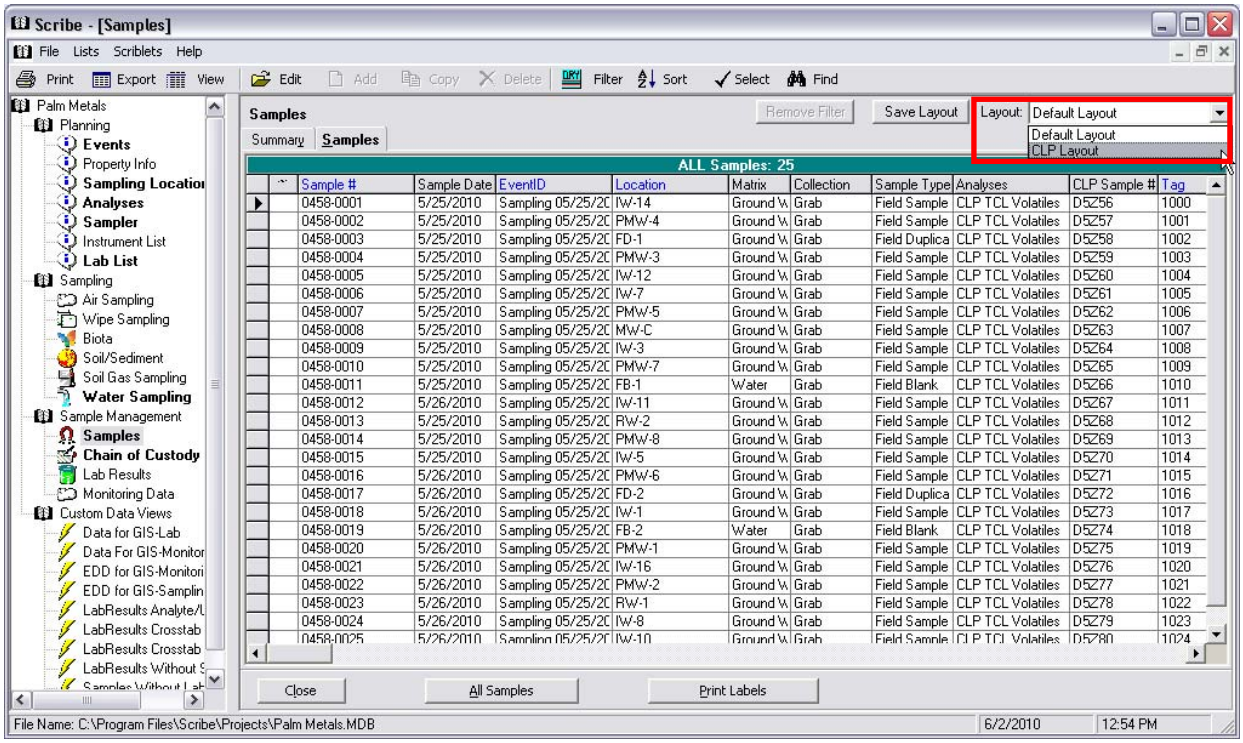
Print Sample Labels

Label options are available through the Samples View. Click on '**Samples**' under Sample Management in the left Navigation Pane. All samples shown on the screen are available to be printed on labels. You can apply Filters, Finds and Sorts to limit the display to the Samples you wish to see.

To configure your labels and print:

1. Click on **drop-down menu** for the Layout field on the top right corner.

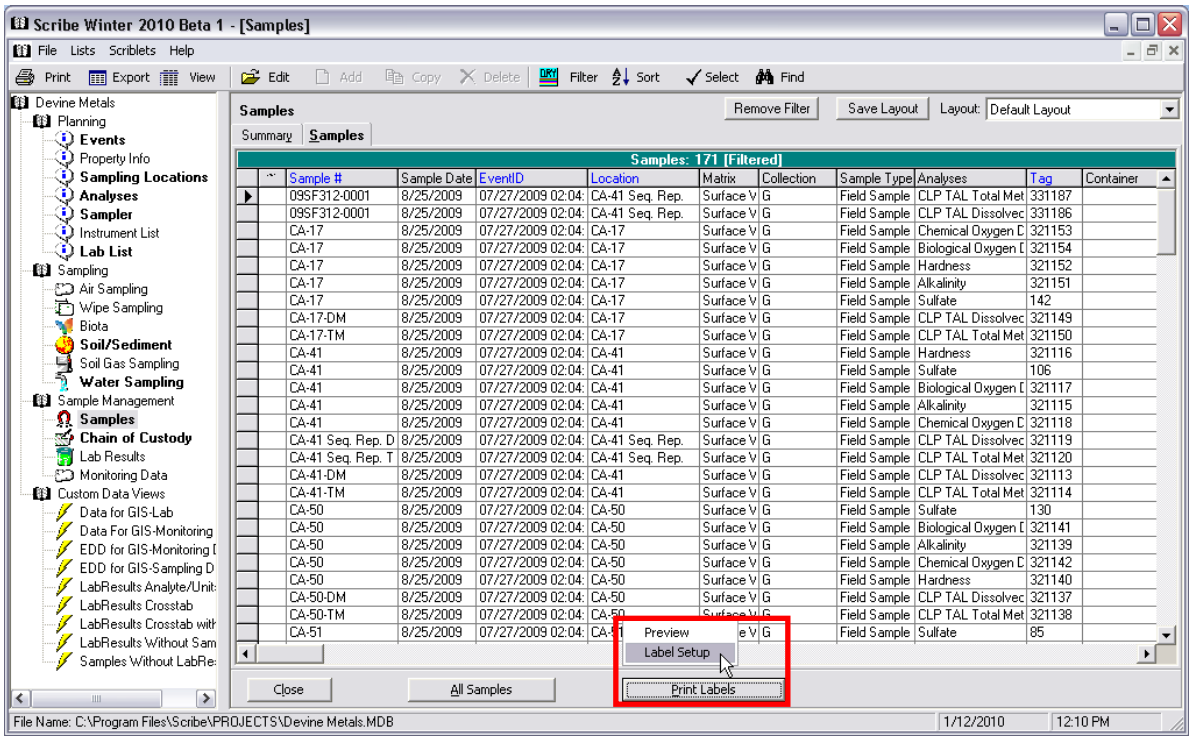
2. Select '**CLP Layout'**. This layout will replace the default Scribe Sample # with the CLP Sample # on the default label layout.

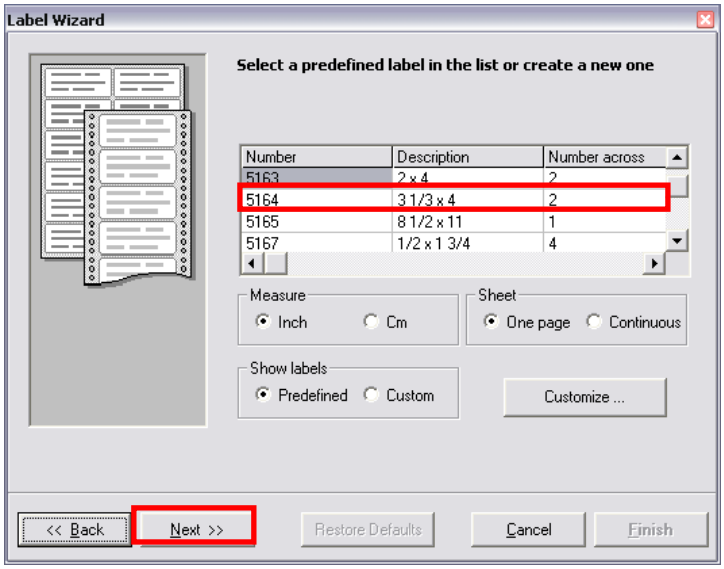


3. Click the '**Print Labels'** button on the bottom of the window.

4. Select '**Label Setup'** if it's the first time you are setting up a label.

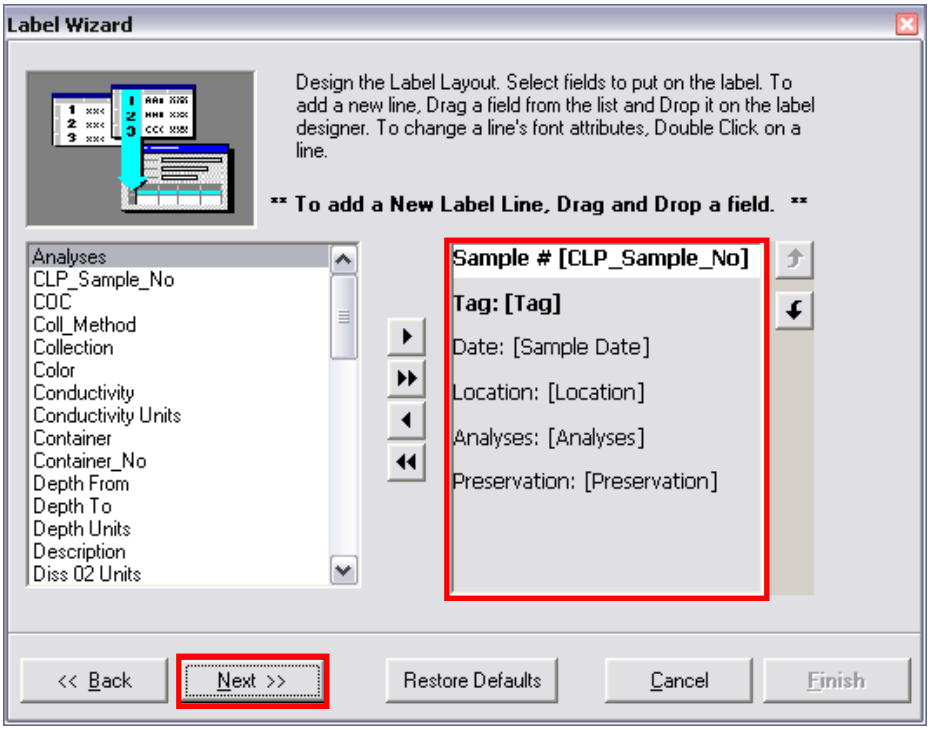
5. Select a pre-defined label format that matches your labels.





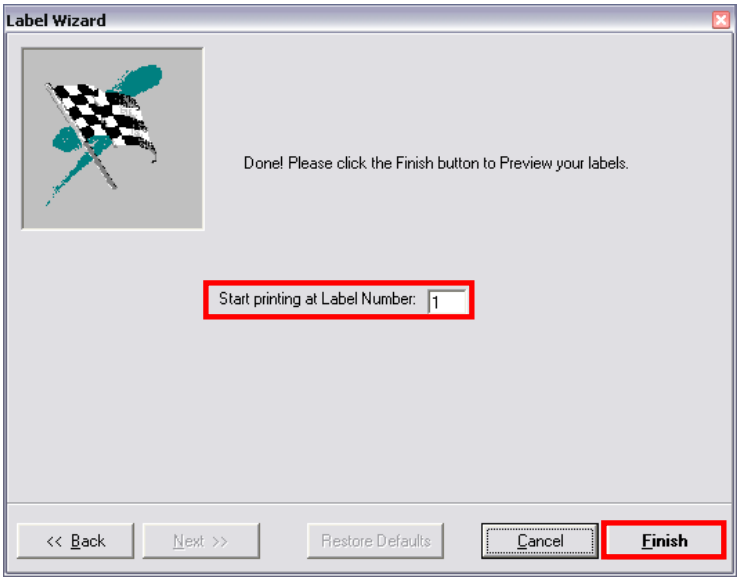
6. Click '**Next**' to continue.

7. Design your label by adding/removing fields to or use the default design. **Note:** The CLP Sample number instead of the Scribe Sample number will be printed on the label.

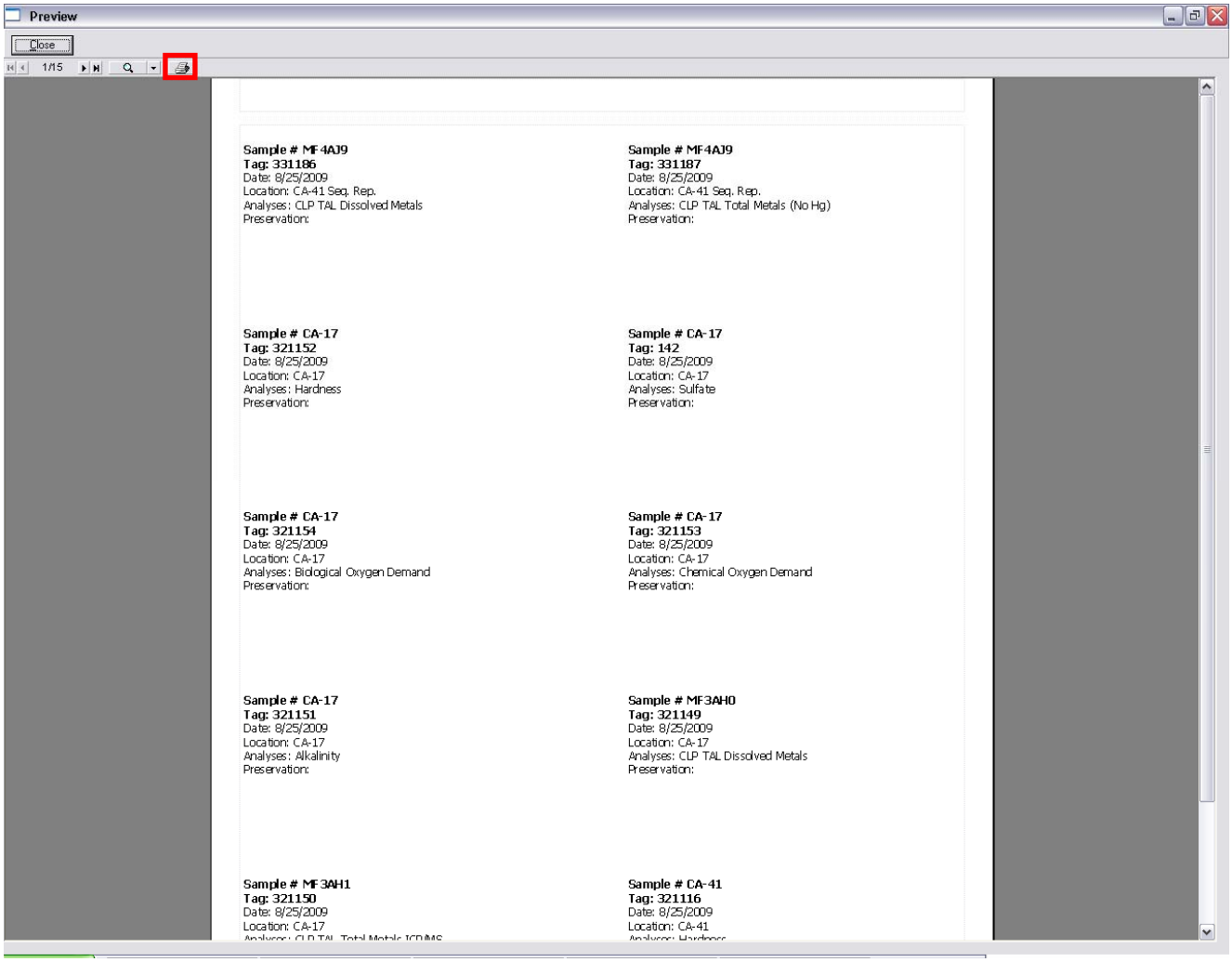


8. Click '**Next**' to continue.

9. If you need to print on half a sheet of labels, use this option to select which label to print on first. Otherwise, click '**Finish**' to continue.



10. A preview of the labels to be printed is displayed.



11. Click on the Printer icon on the top menu bar to print the labels.

Chain of Custody

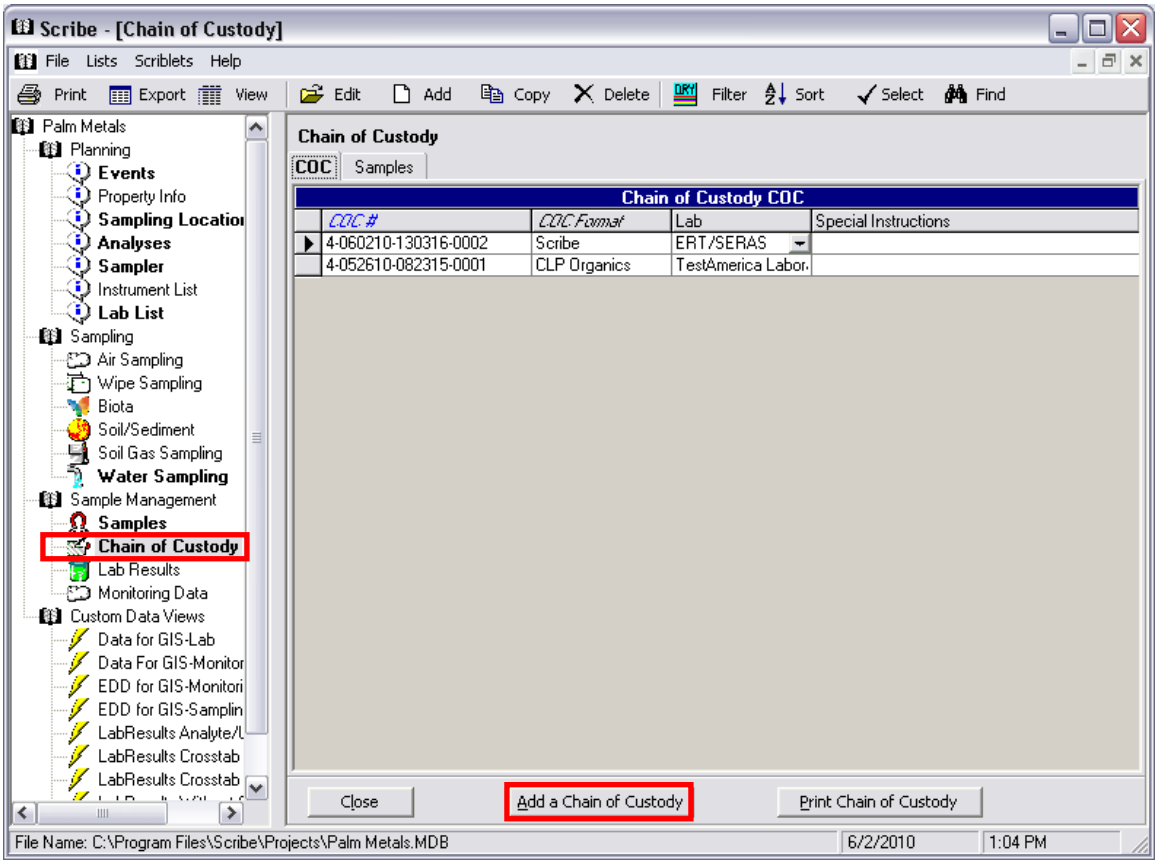
A new feature in Scribe to support CLP sampling is the COC Format for the Chain of Custody. The COC Format option modifies the COC form to adhere to COC standards and requirements. It also controls what samples can be assigned to the COC. For example, Samples with Inorganics analyses can only be assigned to the CLP Inorganics format on the COC.

Note: After submitting samples to the CLP labs, it is recommended that users request the labs to return lab results in electronic format i.e. a spreadsheet (.xls) or a comma-separated text (.csv). Scribe has a Custom Import feature that will import lab result data and marry them up with the sampling data. This effectively eliminates transcription errors and reduces data processing time. See the "Scribe Manual Advanced Part III" for importing details.

Create COC and Assign Samples

To manage and print a Chain of Custody (COC), a COC needs to be created and then samples have to be assigned to the COC:

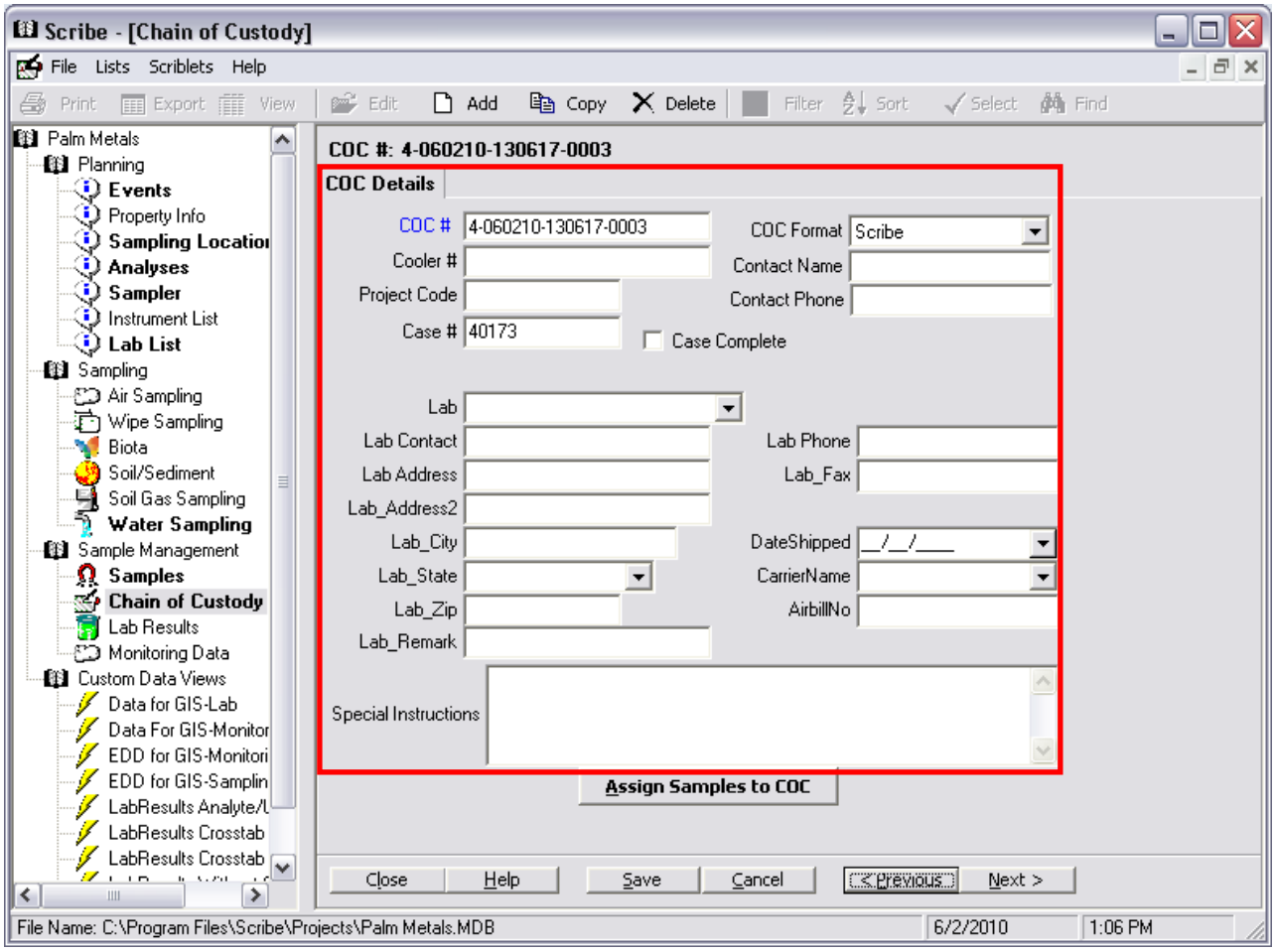
1. Select '**Chain of Custody**' under Sample Management in the left Navigation Pane.



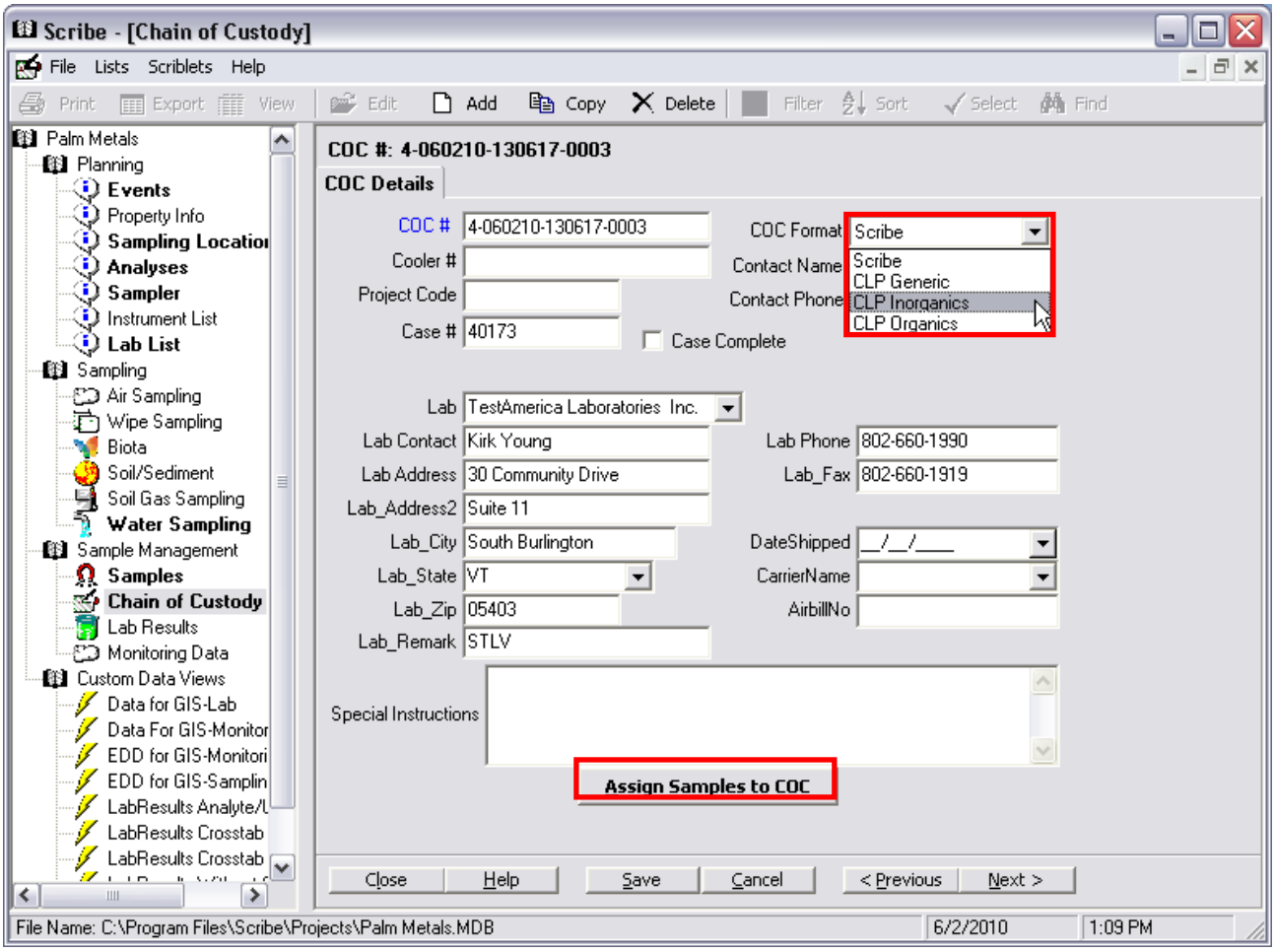
2. Click the '**Add a Chain of Custody**' button on the bottom of the window.

3. The "**COC Details**" screen is displayed.

4. Complete the form by entering other fields such as the **Case #, Cooler #, Lab, and Lab Phone**.

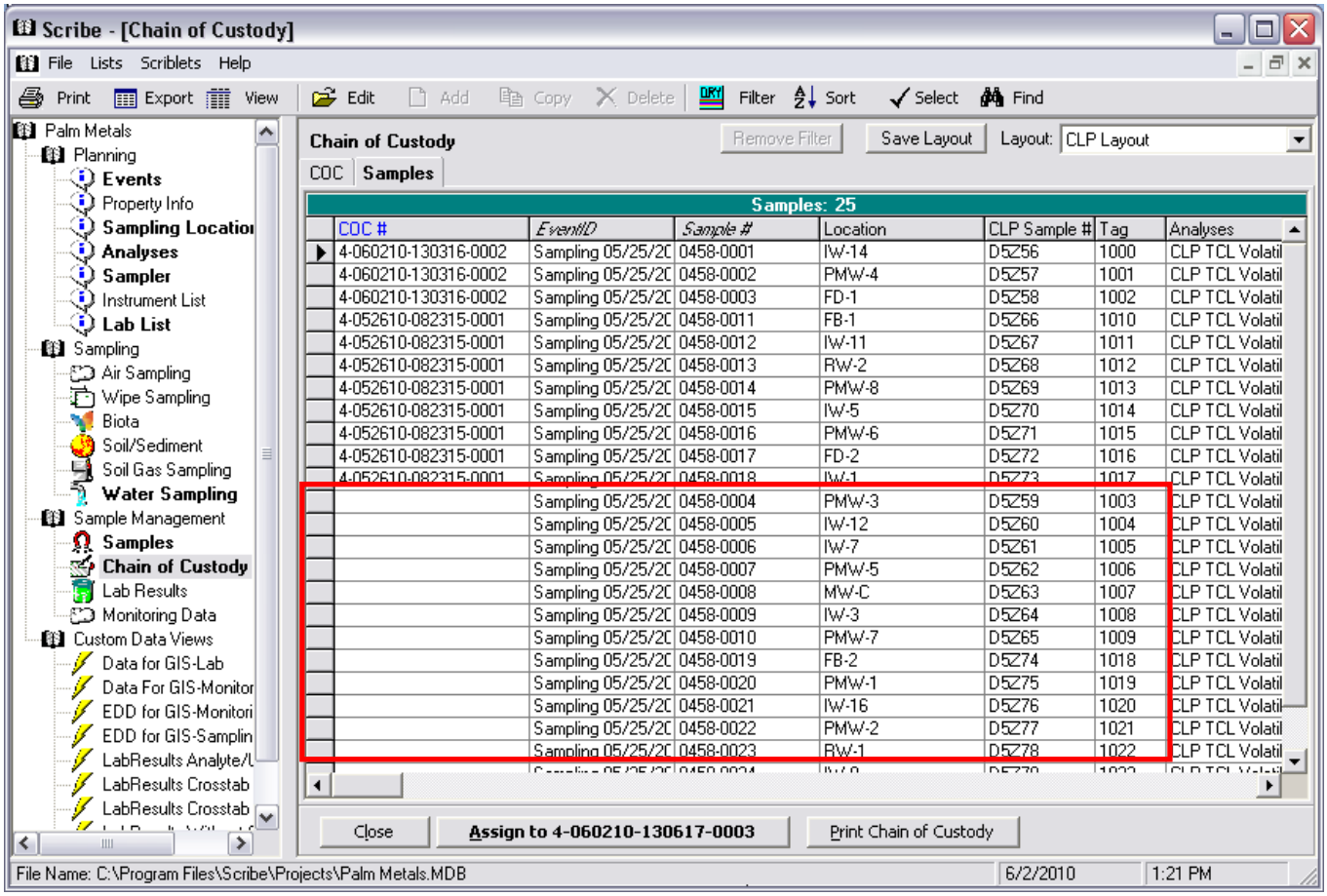


5. Select the appropriate **COC Format** based on the type of COC Samples you are packing. For example, if you are creating a COC for Inorganics, select COC Inorganics. The CLP Generic COC option should be used if you are submitting samples to a program other than CLP but one that requires a CLP/F2L type COC for generating CLP type XML files. Based on the format setting you select, the system will filter for only those types of samples that can be added to this COC.



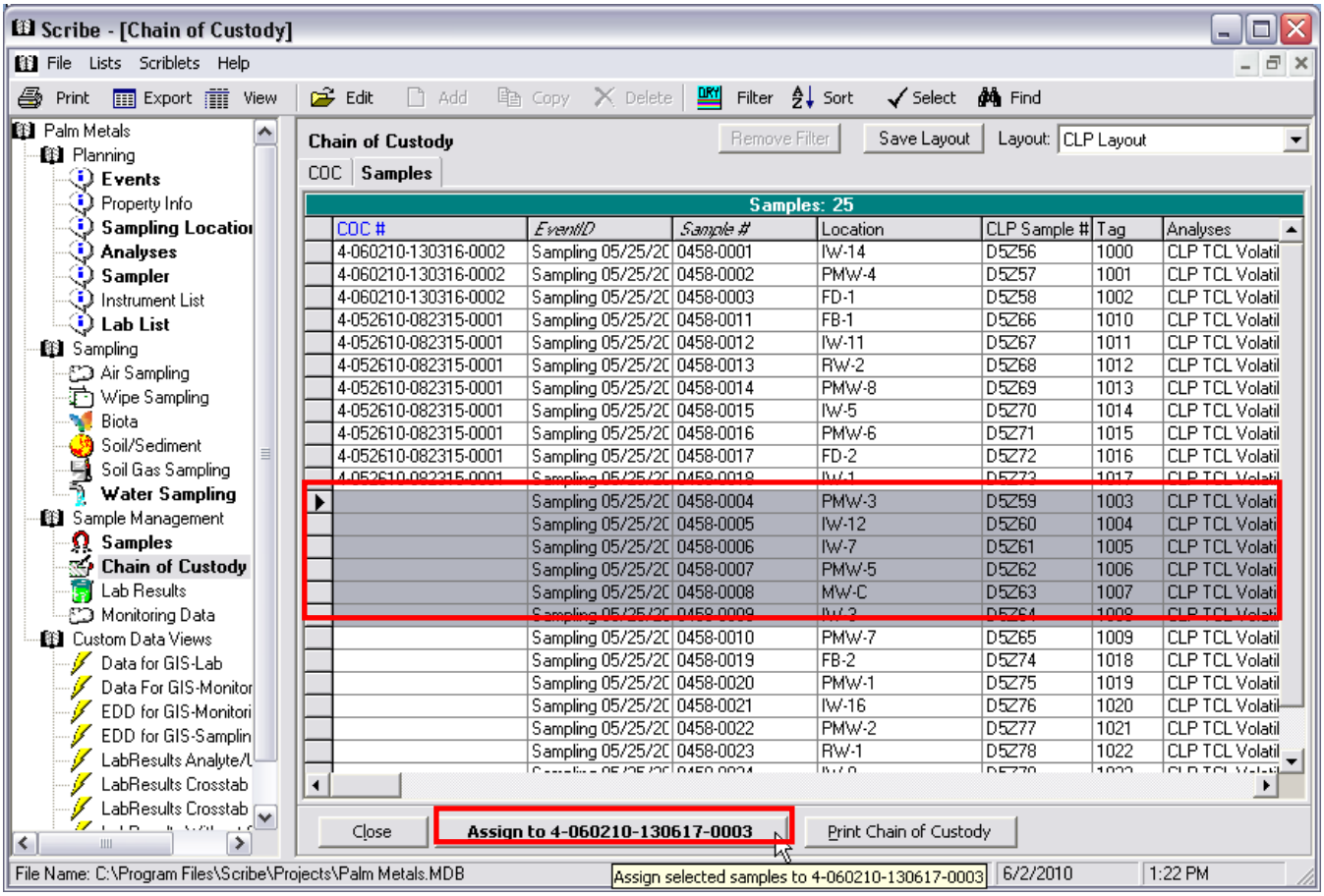
6. Click '**Assign Samples to the COC'** to continue.

7. The "**Chain of Custody Samples**" screen appears. Samples that have not been assigned to a chain are displayed at the bottom of the list.

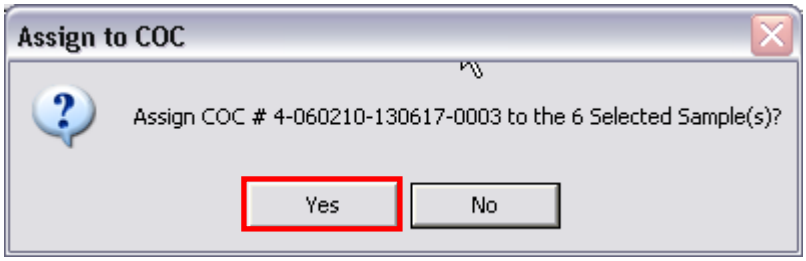


8. Highlight the samples to assign to the new Chain of Custody. Highlight multiple samples by holding down the Shift key or Ctrl key while clicking on the first column before COC# of the samples you wish to assign to the COC.

9. Click the '**Assign to...**' button on the bottom of the window to assign the samples to the Chain of Custody.



10. You will be prompted to confirm. Click '**Yes**' to assign the selected samples to the COC.

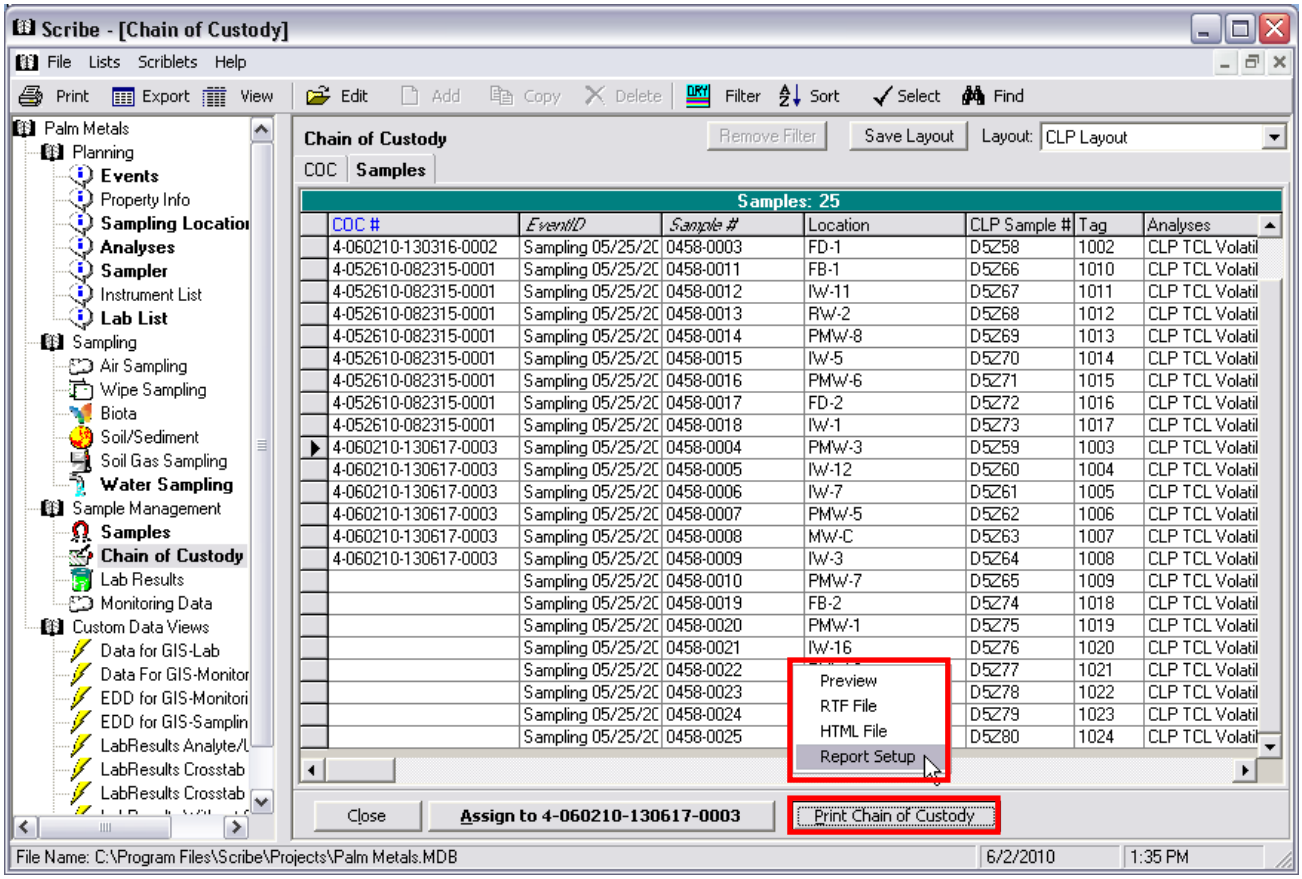


11. You are now ready to configure and print your COC.

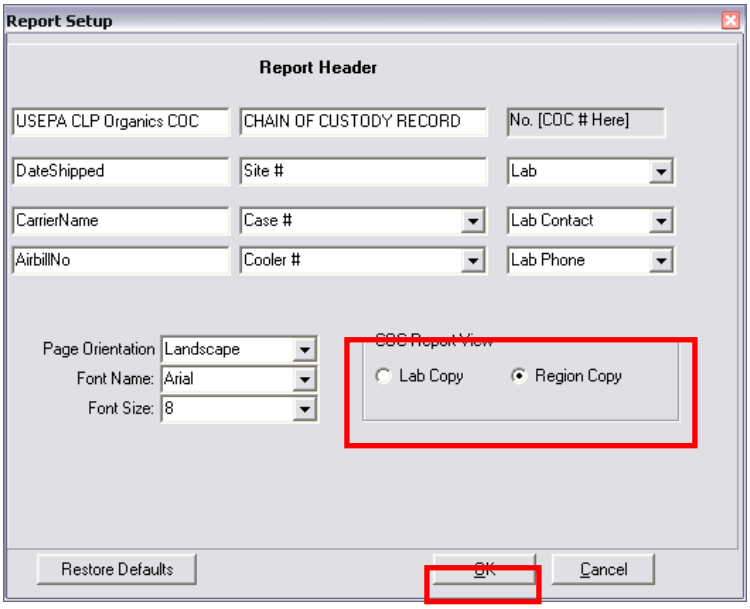
Configure and Print COC

To configure and print a COC:

1. Click the '**Print Chain of Custody**' button.
2. Then select '**Report Setup**'.

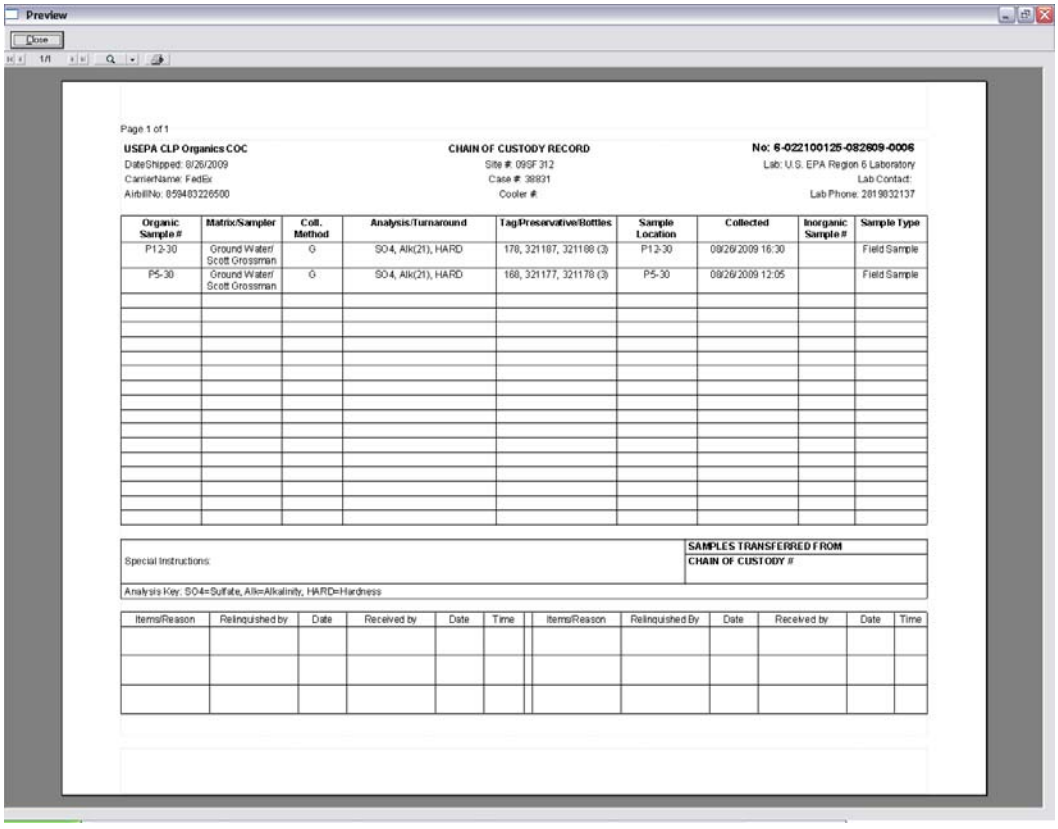


3. The Report Header settings are displayed.



4. The COC Report View (Lab or Region Copy) can also be selected.

5. Click '**OK**' to preview and print the Chain of Custody.

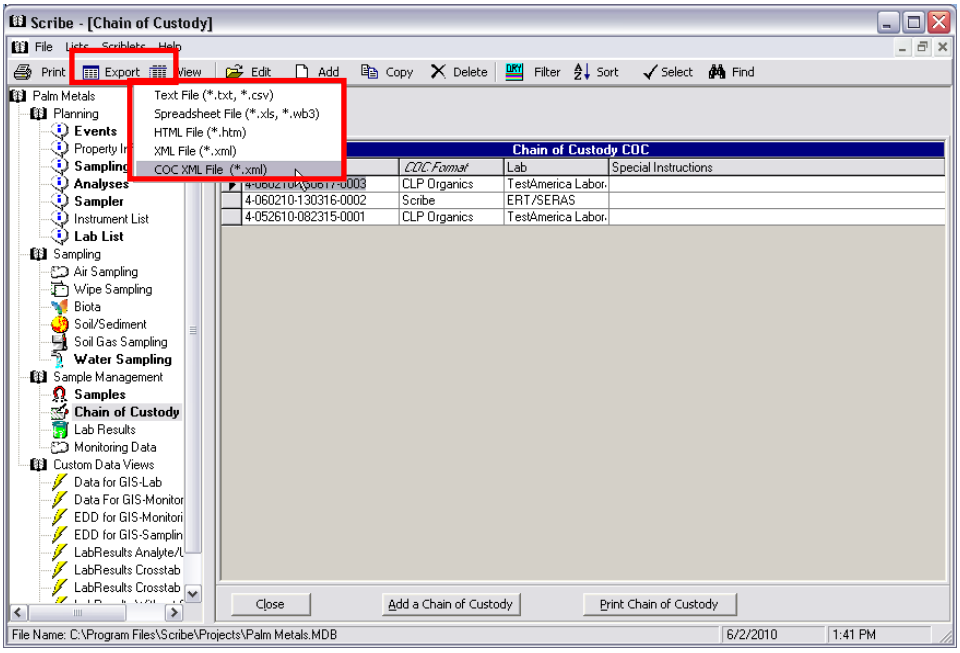


Export to XML File

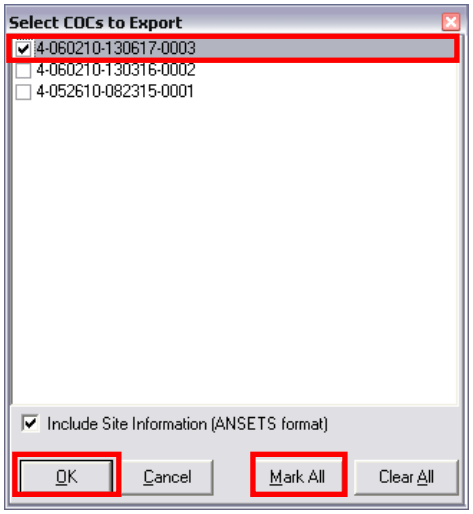
Export COC to XML

A new feature in Scribe is the ability to export the CLP COCs to an XML file. To export:

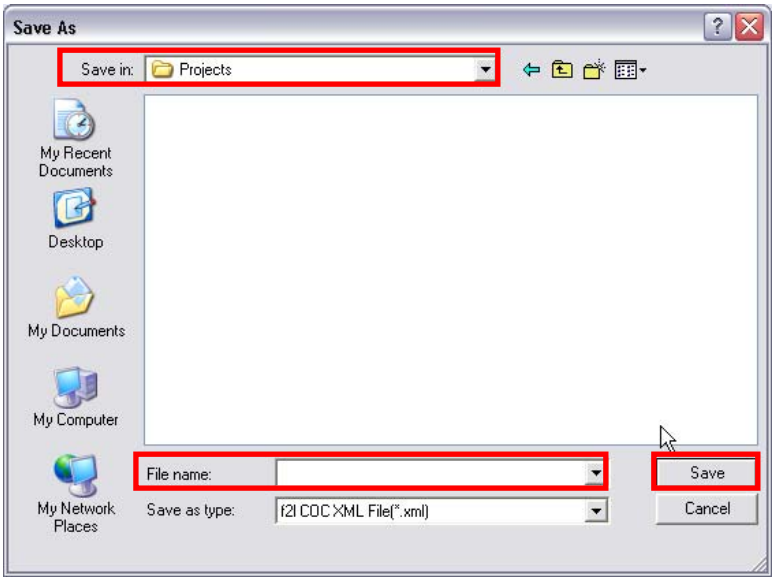
1. Click the '**Export'** button on the top menu bar.
2. Select '**COC XML File (*.xml)**' option.



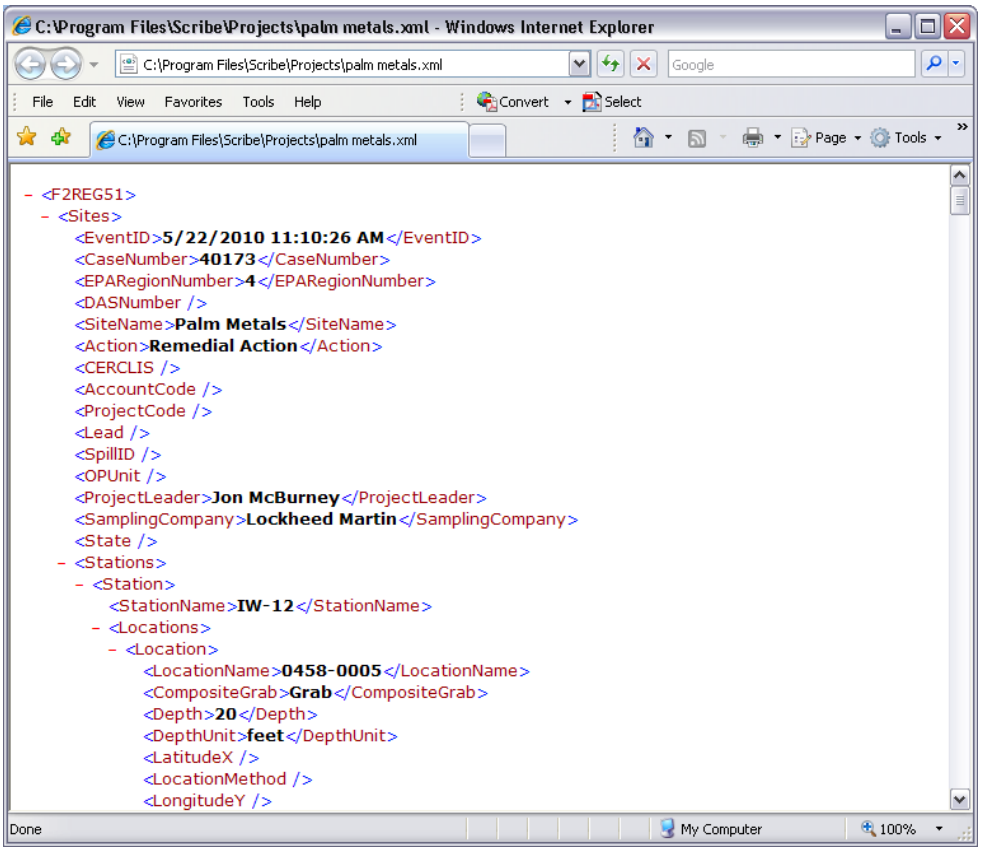
3. Select the Chain of Custody records to export by checking the individual records or click **'Mark All'** to select all COCs.



4. Select your location and provide a filename and click '**Save**'.



5. The XML file will open in Windows Internet Explorer while the file is created and saved.



REPORTING

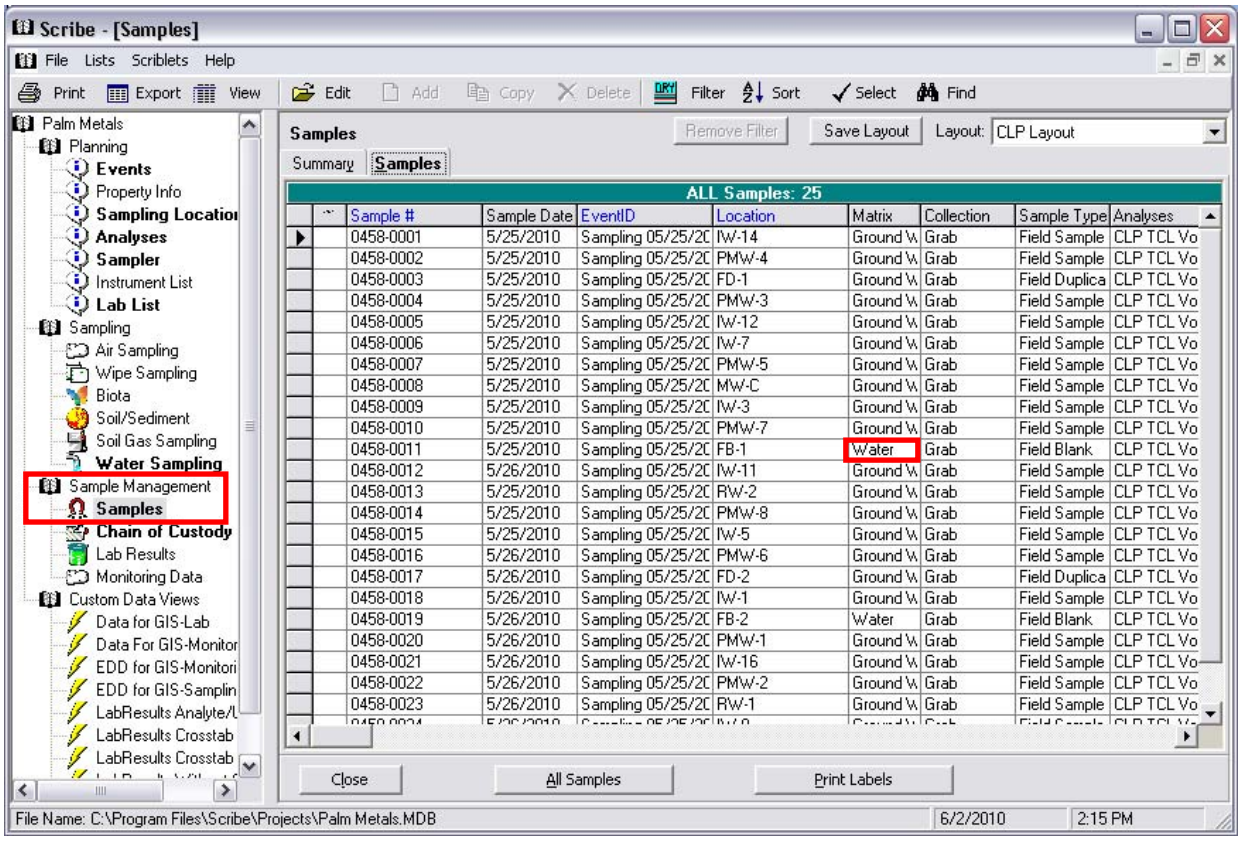
Scribe has flexible reporting options. The most popular way to report out from Scribe is to manipulate the grid view in the All Samples screen to display the data you wish to report. Then export the grid data to an file type that fits your reporting needs. File types include .txt, .csv, .xls, .htm, .xml, .kml, and .kmz.

Find, Filter and Sort

Scribe has built-in user-friendly querying functions such as Find, Filter and Sort. These functions are most useful when you are searching for a particular subset of data that meets one or more criteria.

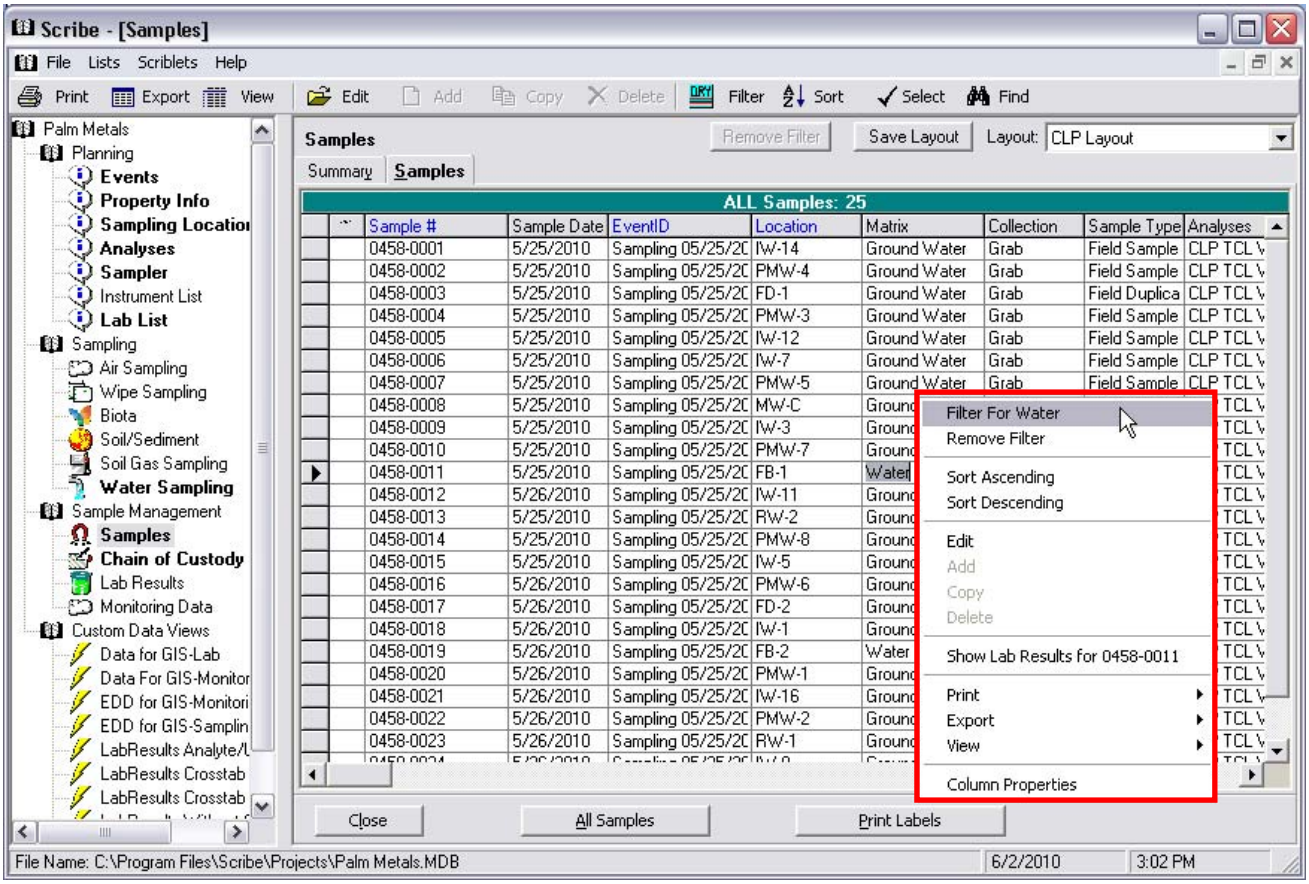
For example, to find and filter for all samples with a Water matrix or Sort ascending/descending:

1. Click on **'Samples'** under Sample Management in the left Navigation bar.



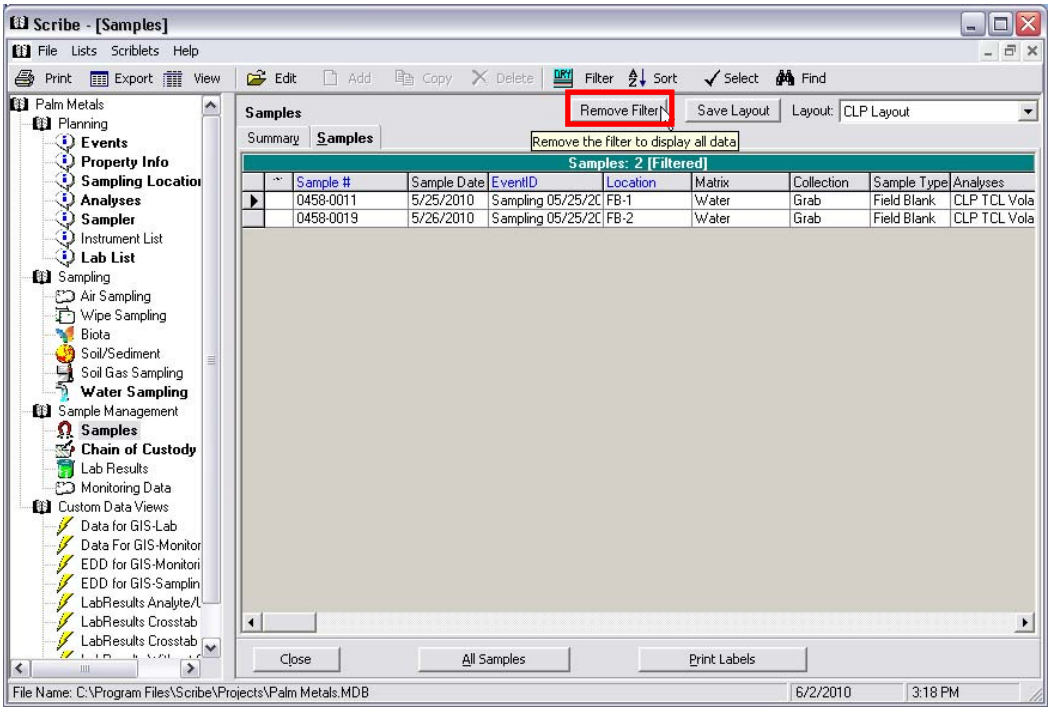
2. To filter or sort on ONE criteria, RT-click on Water value in the Matrix column.

3. Select '**Filter for Water'** in the pop-up menu or select Sort.

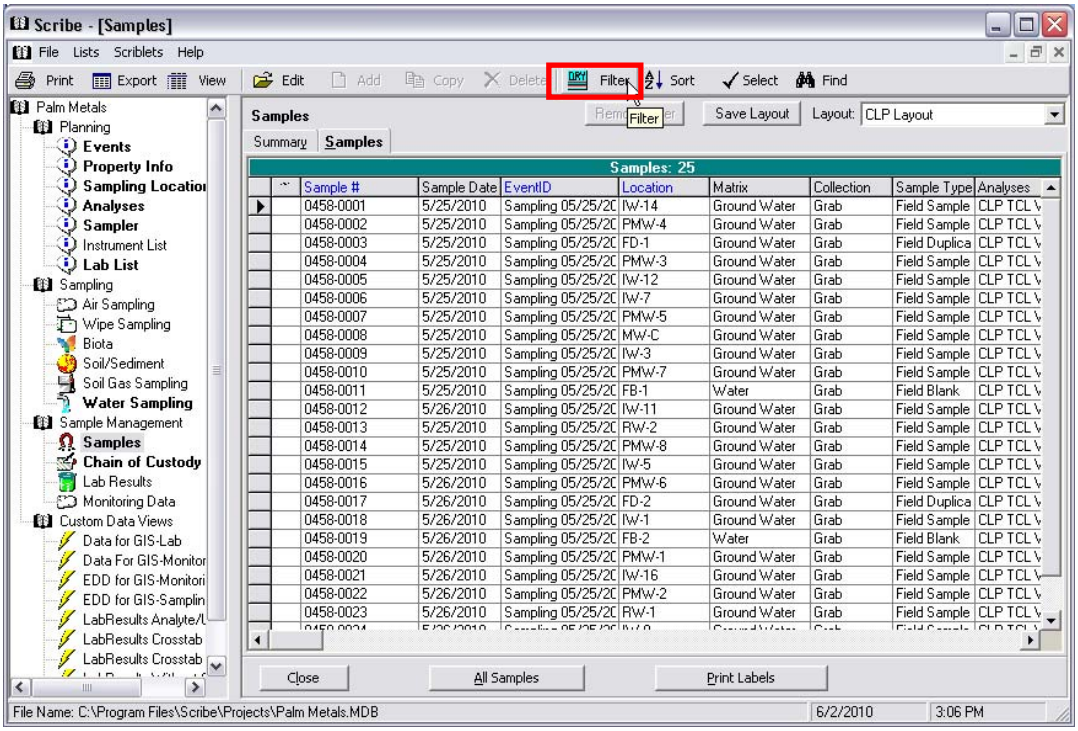


All records that have Water in the Matrix field are displayed.

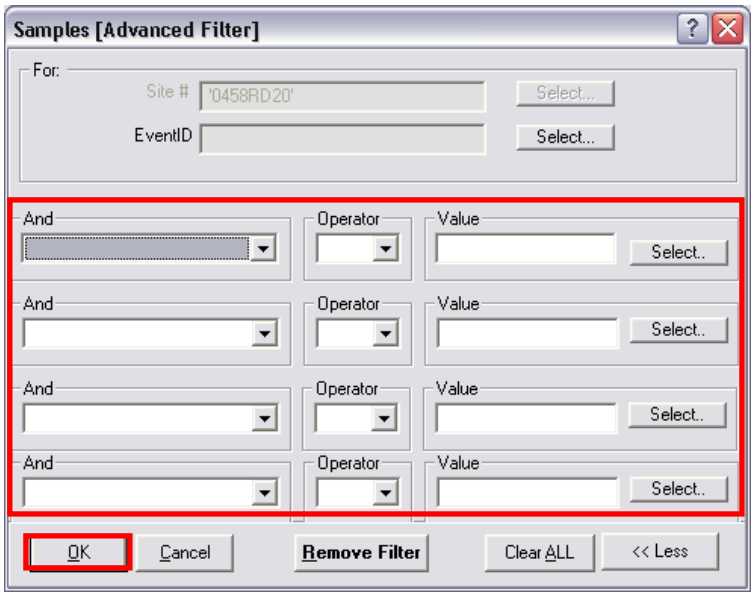
4. To remove the applied filter, click the **'Remove Filter'** button at the top of the screen.



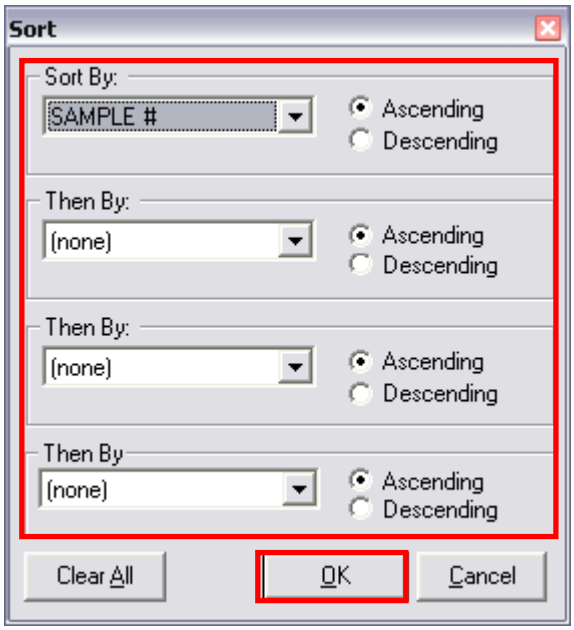
5. To filter on multiple criteria, select the **'Filter'** button on the top menu bar.



6. The Advanced Filter window is displayed. Input the criteria that for your search and click **'OK'** to apply the filter.



7. The Advanced **Sort** button also provides multi-tiered sorting options for sorting on more than one criteria.



Export

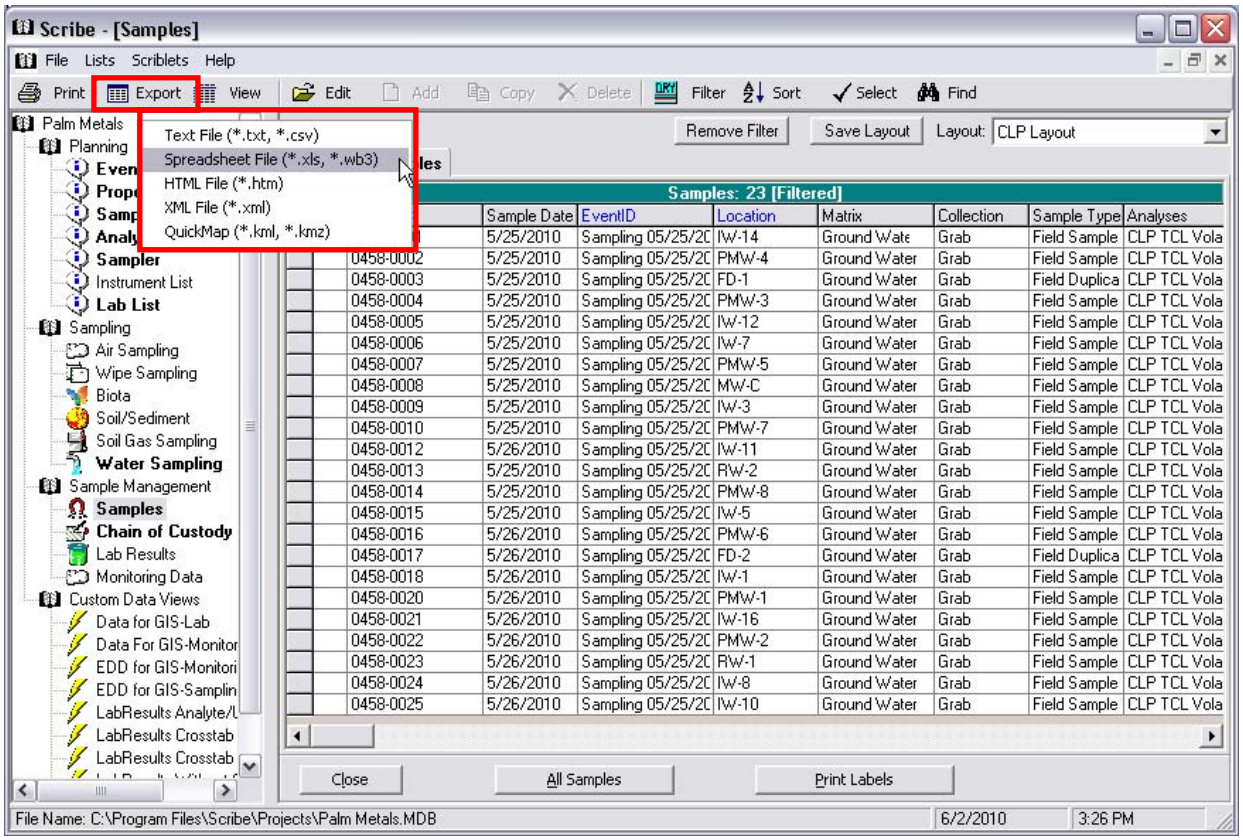
The Scribe grid view does not display every field in Scribe. Select fields are displayed by default and the user can turn on/off the columns. Turn on/off columns as described in the Sample Management section of this document to manipulate the data that is displayed.

After your grid view contains the data necessary for reporting purposes, the user can export the grid view to a third-party file type.

To export the grid view:

1. Click on **'Export'** button on the top menu bar.

2. Select the file type to which you wish to save the data. For example, Spreadsheet (.xls).



3. You will be prompted to select the destination and name the file.

4. The file will open in the external application if it is installed on your computer. For example, if you selected Spreadsheet, Excel will open with the grid data.

Worksheet Reports

Scribe provides a generic worksheet report that allows the user to customize the Header of the report to suit their needs. This option can be used to customize a Samples Report that could be used as a Receipt for Samples on residential sampling tasks.

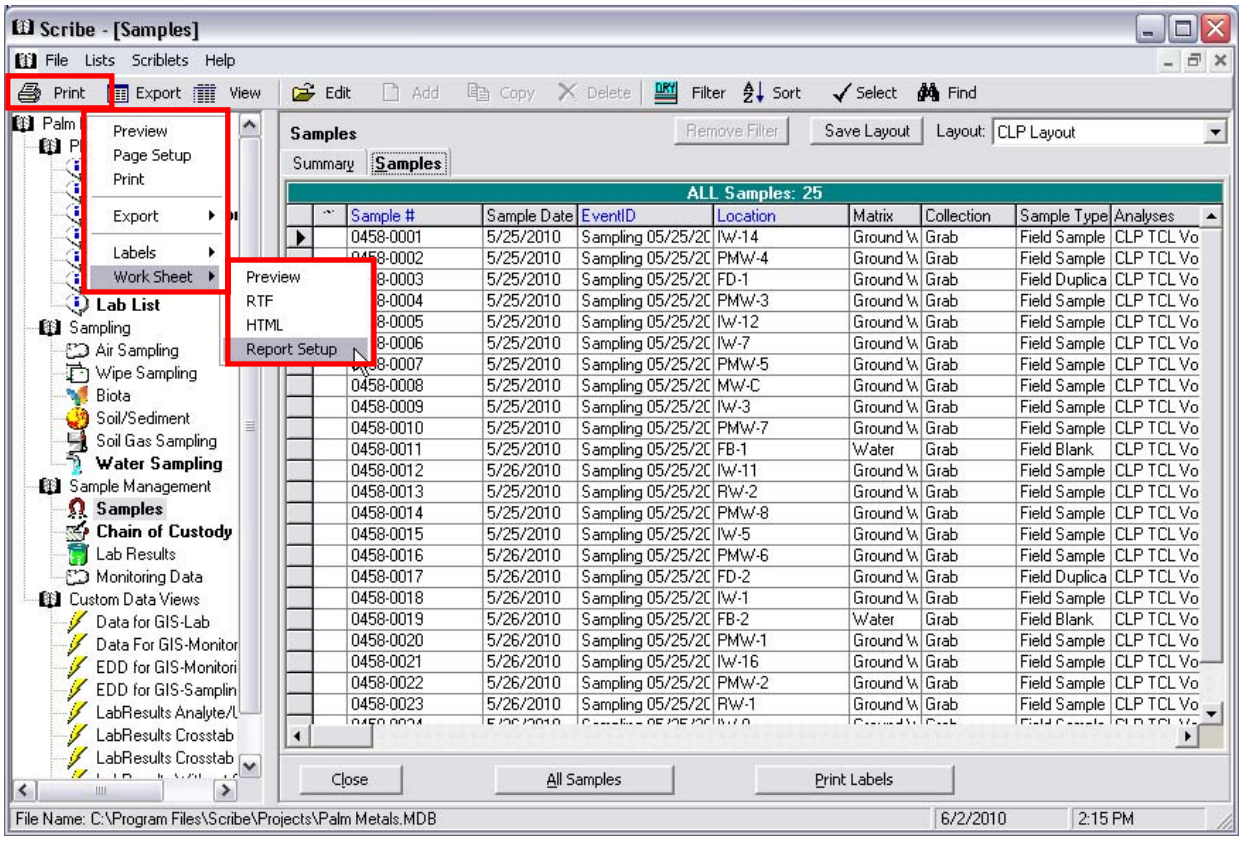
To generate the worksheet report:

1. Use the Find, Filter and Sort options and Column Views to display the data you want to report.

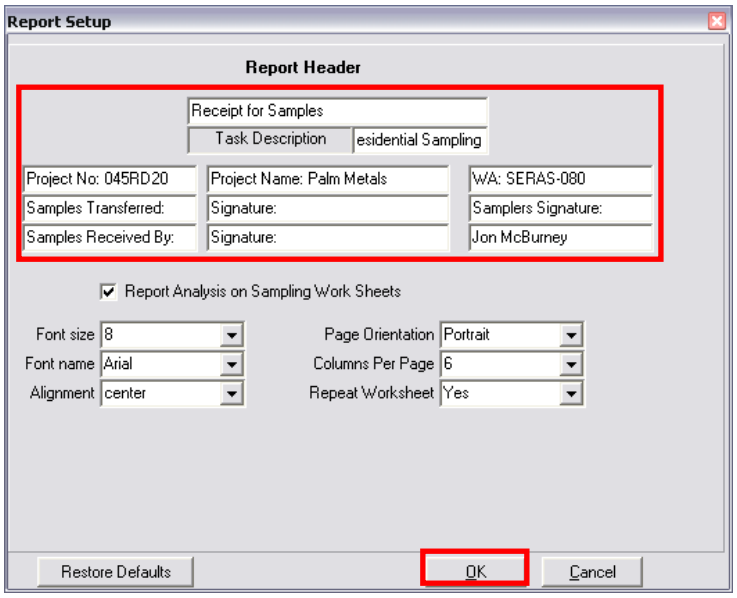
2. Click on the **'Print'** button on the top menu bar.

3. Select the **'Worksheet'** option.

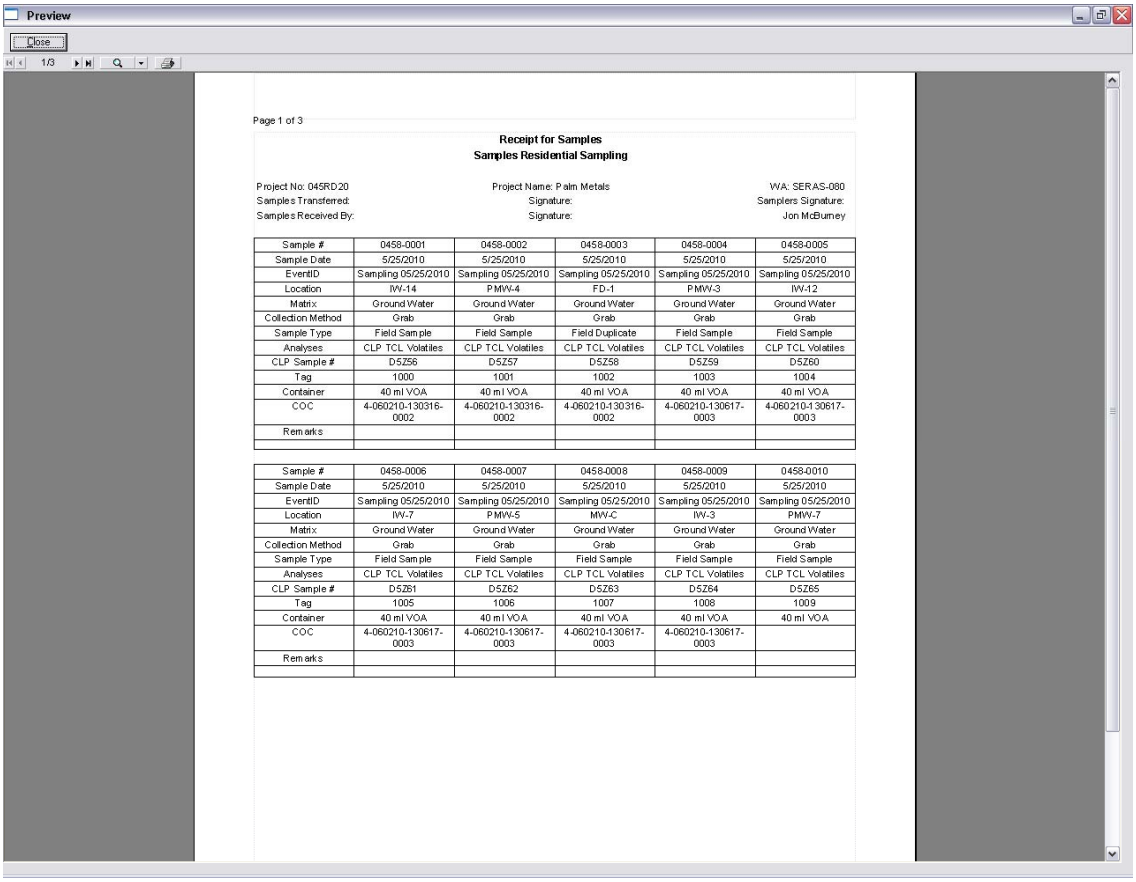
4. Select the **'Report Setup'** option to customize the Header. RTF and HTML will print the worksheet data to the selected format.



5. Configure the Report Header fields to reflect the information that will be displayed at the top of the report.



6. Click **'OK'** and the report is generated.



Sample #	0458-0001	0458-0002	0458-0003	0458-0004	0458-0005
Sample Date	5/25/2010	5/25/2010	5/25/2010	5/25/2010	5/25/2010
EventID	Sampling 05/25/2010	Sampling 05/25/2010	Sampling 05/25/2010	Sampling 05/25/2010	Sampling 05/25/2010
Location	IW-14	PMW-4	FD-1	PMW-3	IW-12
Matrix	Ground Water	Ground Water	Ground Water	Ground Water	Ground Water
Collection Method	Grab	Grab	Grab	Grab	Grab
Sample Type	Field Sample	Field Sample	Field Duplicate	Field Sample	Field Sample
Analyses	CLP TCL Volatiles	CLP TCL Volatiles	CLP TCL Volatiles	CLP TCL Volatiles	CLP TCL Volatiles
CLP Sample #	D5Z56	D5Z57	D5Z58	D5Z59	D5Z60
Tag	1000	1001	1002	1003	1004
Container	40 ml VOA	40 ml VOA	40 ml VOA	40 ml VOA	40 ml VOA
COC	4-060210-130316-0002	4-060210-130316-0002	4-060210-130316-0002	4-060210-130617-0003	4-060210-130617-0003
Remarks					

Sample #	0458-0006	0458-0007	0458-0008	0458-0009	0458-0010
Sample Date	5/25/2010	5/25/2010	5/25/2010	5/25/2010	5/25/2010
EventID	Sampling 05/25/2010	Sampling 05/25/2010	Sampling 05/25/2010	Sampling 05/25/2010	Sampling 05/25/2010
Location	IW-7	PMW-5	MW-C	IW-3	PMW-7
Matrix	Ground Water	Ground Water	Ground Water	Ground Water	Ground Water
Collection Method	Grab	Grab	Grab	Grab	Grab
Sample Type	Field Sample	Field Sample	Field Sample	Field Sample	Field Sample
Analyses	CLP TCL Volatiles	CLP TCL Volatiles	CLP TCL Volatiles	CLP TCL Volatiles	CLP TCL Volatiles
CLP Sample #	D5Z61	D5Z62	D5Z63	D5Z64	D5Z65
Tag	1005	1006	1007	1008	1009
Container	40 ml VOA	40 ml VOA	40 ml VOA	40 ml VOA	40 ml VOA
COC	4-060210-130617-0003	4-060210-130617-0003	4-060210-130617-0003	4-060210-130617-0003	
Remarks					

Field Operating Procedure No. 6 Packing and Shipping of Environmental Samples

Old American Zinc Plant Superfund Site,
Fairmont City, St. Clair County, Illinois
Remedial Design
WA No. 224-RDRD-B5A1/Contract No. EP-S5-06-01

Revision Number: 0
Prepared: 3/10/2017

Approved By:

_____

Rachel Grand, Site Manager Date

_____

Theresa Rojas, Program Quality Manager Date

Packing and Shipping of Environmental Samples

1.1 Purpose

The purpose of this field operating procedure (FOP) is to delineate protocols for the packing and shipping of samples to the laboratory for analysis.

1.2 Scope

This FOP is applicable to all samples collected and prepared for analysis at an offsite laboratory.

1.3 Equipment and Materials

- Waterproof hard plastic coolers
- Plastic zip-top bags
- Plastic garbage bags
- Absorbent packing material (not vermiculite)
- Inert cushioning material (not vermiculite)
- Ice
- EPA Region 5 sample tags
- Scribe software
- Laptop and printer
- Adhesive labels 2 x 4 inches (generated by Scribe software)
- Chain-of-custody forms (generated by Scribe software)
- EPA Region 5 Custody seals
- Airbills and shipping pouches (e.g., FedEx)
- Clear tape
- Strapping tape
- Mailing labels

1.4 Procedures and Guidelines

1.4.1 Prepare Bottles or Bags for Shipment

1. Arrange sample containers in groups by sample number.
2. Check that sample container lids are tight.
3. Secure appropriate EPA Region 5 sample tags around lids of container with string or wire.
4. Arrange containers in front of assigned coolers.
5. Affix appropriate adhesive labels to each container.
6. Enclose each sample in a clear, resealable zip-top bag, making sure that sample labels are visible.

1.4.2 Prepare Coolers for Shipment

1. Tape drains shut, inside and out.
2. Affix “This Side UP” labels on all four sides and “Fragile” labels on at least two sides of each cooler.
3. Place mailing label with laboratory address on top of the coolers.
4. Place inert cushioning material (e.g., bubble wrap, preformed poly-foam liner) in the bottom of the cooler. Do not use vermiculite.
5. Place appropriate chain-of-custody records with corresponding custody seals on top of each cooler.
6. Double-bag and seal loose ice in resealable, plastic, zip-top bags to prevent melting ice from leaking and soaking the packing material. Place the ice outside the garbage bags containing the samples. Place sufficient ice in cooler to maintain the internal temperature at 4±2 degrees Celsius during transport.
7. Put an absorbent pad in the bottom of the cooler and fill the cooler with enough packing material to prevent breakage of the sample bottles and to absorb the entire volume of the liquid being shipped (offsite sample shipment only).
8. Record the EPA Region 5 custody seals on the chain-of-custody forms. Sign each chain-of-custody form (or obtain signature) and indicate the time and date the cooler was custody sealed.
9. Seal the laboratory copies of the chain-of-custody forms in a large resealable plastic zip-top bag and tape to the inside lid of the cooler. Retain the Region 5 copies of the chain-of-custody forms for return to EPA. Each cooler must contain a chain-of-custody form (or forms) that corresponds to the contents of the cooler.
10. Close lid and latch.
11. Peel custody seals carefully from backings and place intact over lid openings (right front and left back). Cover seals with clear protection tape.
12. Tape cooler shut on both ends, making several complete revolutions with strapping tape. **Do not** cover custody seals.
13. Relinquish to carrier (e.g., FedEx). Place airbill receipt inside the mailing envelope and send to sample documentation coordinator, along with the other documentation.

1.5 Attachments

None.

1.6 Key Checks

- Ensure completeness of the airbill.
- Verify that the shipment is not leaking from wet ice.

1.7 References

None.

Field Operating Procedure No. 7
Note Taking and Field Logbook

Old American Zinc Plant Superfund Site,
Fairmont City, St. Clair County, Illinois
Remedial Design
WA No. 224-RDRD-B5A1/Contract No. EP-S5-06-01

Revision Number: 0
Prepared: 3/10/2017

Approved By:

_____

Rachel Grand, Site Manager Date

_____

Theresa Rojas, Program Quality Manager Date

Note Taking and Field Logbook

1.1 Purpose

The purpose of this field operating procedure (FOP) is to delineate protocols for recording field and sampling information in a field logbook.

1.2 Scope

Data generated from the use of this FOP may be used to support the following activities: site characterization, remedial investigation, and predesign sampling.

1.3 Equipment and Materials

- Field logbook
- Indelible black ink pen
- Write-in-the-rain pen (for extreme weather conditions—cold/rain)

1.4 Procedures and Guidelines

All information pertinent to a field or sampling effort will be recorded in a bound field logbook that will be initiated at the start of the first onsite activity. The field logbook will consist of a bound notebook with consecutively numbered pages that cannot be removed. The outside front cover of the logbook will contain the project (site) name and the specific activity (e.g., supplemental remedial investigation). The inside front cover will include the following:

- Site name and EPA work assignment number
- Project number
- Site manager’s name and mailing address
- Sequential logbook number
- Start date and end date of logbook

Each page will be consecutively numbered, dated, and initialed. All entries will be made in indelible black ink, and all corrections will consist of line-out deletions that are initialed and dated. If only part of a page is used, then the remainder of the page should have an "X" drawn across it. At a minimum, entries in the logbook will include the following:

- Time of arrival and departure of site personnel, site visitors, and equipment
- Instrument calibration information, including make, model, and serial number of the equipment calibrated
- Description of significant activities for the day
- Documentation of photographs taken during field activities (e.g., date, time, and description of photograph)
- Field observations (e.g., sample description, weather, unusual site conditions or observations, sources of potential contamination, etc.)
- Detailed description of the sampling location, including a sketch when necessary

- Details of the sample site (e.g., coordinates [x, y], water elevation [z], casing diameter and depth, integrity of the casing, etc.)
- Sampling methodology and matrix, including distinction between grab and composite samples
- Names of field team members and subcontractors
- Start or completion time of sample collection activities
- Field measurements (e.g., water depths, sediment probe depths)
- Type of sample (e.g., sediment, groundwater, surface water, soil, debris)
- Number, depth, and volume of sample collected
- Field sample number
- Requested analytical determinations
- Sample preservation
- Quality control samples associated with the sample
- Sample shipment information including chain-of-custody form number and laboratory, carrier, date, and time
- Health and safety issues (including level of personal protective equipment)
- Signature and date by personnel responsible for observations

Sampling situations vary widely. No general rules can specify the extent of information that must be entered in a logbook. However, records should contain sufficient information so that someone can reconstruct the sampling activity without relying on the collector's memory. The field team leader will keep a master list of all field logbooks assigned to the sampling crew.

1.5 Attachments

None.

1.6 Key Checks and Items

None.

Field Operating Procedure No. 8
Equipment Decontamination Procedures

Old American Zinc Plant Superfund Site,
Fairmont City, St. Clair County, Illinois
Remedial Design
WA No. 224-RDRD-B5A1/Contract No. EP-S5-06-01

Revision Number: 0
Prepared: 3/10/2017

Approved By:

_____

Rachel Grand, Site Manager Date

_____

Theresa Rojas, Program Quality Manager Date

Equipment Decontamination Procedures

1.1 Purpose

This field operating procedure (FOP) provides updated guidelines for the equipment decontamination procedures to be implemented during soil sampling at the Old American Zinc Plant Superfund site in Fairmont, Illinois.

1.2 Equipment and Materials

- Distilled water
- Liquinox detergent
- Two 1-gallon sprayers (1 filled with distilled water, 1 filled with 1 percent Liquinox solution)
- Nitrile gloves
- Paper towels

1.3 Procedure: Sampling Equipment Decontamination—Hand Augers, Trowels, and Drill Rig Core Barrels

Decontamination procedures will be conducted in accordance with the following guidelines:

1. Wear unpowdered chemical-resistant nitrile gloves.
2. Make a solution of approximately **1 gallon** of distilled water and **2.5 tablespoons of Liquinox** (for 1 percent solution) in a 1½-gallon sprayer.
3. Remove gross contamination from the sampling tool at site of sampling (sample hole).
4. After **each** sample is collected (from each aliquot in one sample area/ depth), the sampling tool will be decontaminated using a spray bottle of Liquinox solution. After gross contamination is removed, liberally spray the sampling tool with the solution over the ground surface. The overspray can be allowed to disperse into the yard onto the grassy or soil surface. **Do not spray over the sampling borehole location.** Spray the sampling tool (inside and out) and handle (i.e., any surfaces that came into contact with the potentially contaminated soil)**.**
5. Prior to rinsing, use a clean paper towel to wipe off excess dirt and soapy spray. Discard used paper towels with other used sampling equipment and personal protective equipment.
6. Rinse the sampling tool using the clean distilled water spray. The overspray can be allowed to disperse into the yard onto the grassy or soil surface. Do not spray over the sampling borehole location.
7. Completely air dry the sampling tool or wipe dry with a clean paper towel.
8. For **each property,** document decontamination by indicating the decontamination was conducted in accordance with this FOP.

Note: If residents have questions regarding the overspray procedure, inform them that the Liquinox (and Liquinox solution) is a biodegradable, phosphate-free detergent and should not adversely affect their grass. Liquinox safety data sheet is available and attached to this document.

1.4 Attachments

Liquinox Safety Data Sheet.

1.5 Key Checks

1. Do not use acetone for decontamination.
2. Clean with solutions of Liquinox or equivalent phosphate-free detergent, and distilled water.

1.6 References

None.

ALCONOX

Critical-cleaning detergents for laboratory, healthcare and industrial applications

30 Glenn Street
White Plains NY 10603 USA
Tel.914.948.4040
Fax.914.948.4088
Toll Free 877-877-2526

24 Hour Emergency Number (CHEM-TEL) (800) 255-3924 in US or ++813-248-0513

(e-mail) cleaning@alconox.com

(URL) http://www.alconox.com
- **distributors**
- **technical information**
- **free samples**
- **new developments**

Liquinox®

Critical-Cleaning Liquid Detergent

- Concentrated to save you money
- Replaces corrosive acids and hazardous solvents
- Phosphate free, biodegradable and readily disposable
- Free rinsing to give you reliable results and no interfering residues
- Use to pass your cleaning validation tests for lab accreditation and plant inspection approval

Used to clean: Healthcare instruments, laboratory ware, vacuum equipment, tissue culture ware, personal protective equipment, sampling apparatus, catheters, tubing, wine glasses, clean rooms, medical devices, optical parts, electronic components, pharmaceutical apparatus, cosmetics manufacturing equipment, metal castings, forgings and stampings, industrial parts, pipes, tanks and reactors. Authorized by USDA for use in federally inspected meat and poultry plants. Passes inhibitory residue test for water analysis. Used for phosphate sensitive analysis ware. FDA certified.

Used to remove: Soil, grit, grime, slime, grease, oils, blood, tissue, particulates, deposits, chemical and solvents.

Surfaces cleaned: Corrosion inhibited formulation recommended for glass, metal, stainless steel, porcelain, ceramic, plastic, cement and fiber glass. Can be used on soft metals such as copper, aluminum, zinc and magnesium if rinsed promptly. Used for art restoration. Corrosion testing may be advisable.

Cleaning method: Soak, brush, sponge, cloth, ultrasonic, flow through clean-in-place. Will foam—not for spray or machine use.

Directions: Make a fresh 1% solution (2 1/2 Tbsp. per gal., 1 1/4 oz. per gal. or 10 ml per liter) in cold, warm or hot water. If available, use warm water. Use cold water for blood stains. For difficult soils, raise water temperature and use more detergent. Clean by soak, circulate, wipe or ultrasonic method. Not for spray machines, will foam. RINSE THOROUGHLY—preferably with running water. For critical cleaning, do final or all rinsing in distilled, deionized or purified water. For food contact surfaces, rinse with potable water. Used on a wide range of glass, ceramic, plastic and metal surfaces. Corrosion testing may be advisable.

Available in convenient sizes:	**Alconox Catalog #**
Case 12 x 1 quarts	**1232**
Case of 4 x 1 gallons	**1201**
15 gallon drum	**1215**
30 gallon drum	**1230**
55 gallon drum	**1255**
1 gallon of concentrate makes 100 gallons of cleaning solution	

Liquinox is available from leading laboratory, hospital, clinical and industrial suppliers. To find a distributor for Alconox, Inc. detergents, visit “Find Dealer” at the website. To request FREE samples, visit Sample Request at www .alconox.com, write or call Alconox, Inc. today

MS 02.40.01.16.98.1

Critical-cleaning detergents for laboratory, healthcare and industrial applications

30 Glenn Street
White Plains NY 10603 USA
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Fax.914.948.4088
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(e-mail) cleaning@alconox.com

(URL) http://www.alconox.com
- **distributors**
- **technical information**
- **free samples**
- **new developments**

PHYSICAL DATA	Typical Value
pH as is	8.5
Specific gravity (g/ml)	1.07
Density (lbs./gal.)	8.9
Vapor pressure (mm Hg)	10.5
Flash Point (degrees F)	None
Phosphate Content (as Phosphorus)	0%
Fragrance and Dye Content	0%
Color:	Pale Yellow
Form:	Liquid
Solubility in Water:	Completely soluble in all preparations
Hard Water Effectiveness:	Highly Effective
Biodegradability:	Biodegradable
Foam Tendency:	High Foaming
Shelf Life:	Two years from month of manufacture

Chemical Description

Liquinox consists primarily of a homogeneous blend of sodium linear alkylaryl sulfonate, sodium xylene sulfonate, and ethoxylated alcohol. Liquinox is anionic in nature.

Cleaning Validation Methods:

Test a parameter of rinse water before and after rinsing the cleaned surface, or test the clean surface. No significant change in the parameter indicates no detectable residue. Parameters measured include: pH, conductivity, UV, TOC, HPLC, sodium concentration, phosphorus concentration, anionic surfactant concentration using inexpensive detergent water testing kits, surface tension, and surface analysis. For details see the Pharmaceutical Cleaning Validation References at www.alconox.com

Health Safety Information:

OSHA Hazardous Ingredients: None
RCRA Hazard Class: Non-hazardous
RoHS: No RoHS hazardous ingredients
EPA Priority Pollutants: None
DOT Hazard Class: Non-hazardous
Flammability: Non-flammable
Latex Content: None in detergent, packaging materials or adhesives.
Oral Toxicity: LD_{50} >5000 mg/kg (Rat, Oral)
Eye Irritation: Mild to Moderate eye irritant if not rinsed
Inhalation Toxicity: Non-irritating
VOC Content: 0%
Carcinogenicity: NTP = No **IARC** = No **OSHA** = No
All ingredients in Liquinox are listed in TSCA inventory.
Precautions:
No special precautions other than good industrial hygiene and safety practices employed with any industrial chemical (see Directions). A Material Safety Data Sheet is available at www.alconox.com or by calling 914-948-4040 and asking for the SDS. prompts. The Liquinox catalog number starts with 12.
Contact Alconox, Inc. for purchase specifications. Information presented is typical. Not to be taken as specifications. Typical data is not a specification

Appendix B
Analytical Standard Operating Procedures

METHOD 1340

IN VITRO BIOACCESSIBILITY ASSAY FOR LEAD IN SOIL

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts formally trained in the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique, which a laboratory can use as a basic starting point for generating its own detailed standard operating procedure (SOP), either for its own general use or for a specific project application. Performance data included in this method are for guidance purposes only and must not be used as absolute quality control (QC) acceptance criteria for the purposes of laboratory QC or accreditation.

1.0 SCOPE AND APPLICATION

1.1 The purpose of this method is to define the proper analytical procedure for the validated *in vitro* bioaccessibility (IVBA) assay for lead in soil, to describe the typical working range and limits of the assay, quality assurance (QA), and to indicate potential interferences. At this time, this method has only been validated for lead-contaminated soil under field conditions and not for other matrices (e.g., water, air, amended soils, dust, food, etc.).

1.2 This method is typically applicable for the characterization of lead bioaccessibility in lead-contaminated soil under field conditions. Users are cautioned that deviations in the method from the assay as described may impact the results and the validity of the method. Users are strongly encouraged to document any deviations, as well as any comparisons with other methods and associated QA in any report.

1.3 It is not recommended to analyze IVBA for soils exceeding a total lead concentration of 50,000 mg/kg in order to avoid saturation of the extraction fluid and because risk management decisions are not likely to be improved by analyzing IVBA for soil with concentrations of lead above this level.

1.4 Knowledge of lead bioavailability is important because the amount of lead that actually enters the blood and body tissues from an ingested medium depends on the physical-chemical properties of the lead and of the medium. For example, lead in soil may exist, at least in part, as poorly water-soluble minerals, and may also exist inside particles of inert matrices such as rock or slag of variable size, shape, and association. These chemical and physical properties may tend to influence (usually decrease) the absorption (bioavailability) of lead when ingested. Thus, equal ingested doses of different forms of lead in different media may not be of equal health concern. For more information, see Reference 13.

1.5 Prior to employing this method, analysts are advised to consult the base method for each type of procedure that may be employed in the overall analysis (e.g., Methods 9040 and 9045 for pH and Methods 6010, 6020, and 6800 for determinative methods for the target analytes) for additional information on QC procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts should also consult the disclaimer statement at the front of the manual and the information in Chapter Two for: 1) guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies; and

2) the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by the Environmental Protection Agency (EPA) as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives (DQOs) for the intended application.

1.6 This method is restricted to use by, or under supervision of, properly experienced and trained personnel. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

After drying and sieving, 1 g of soil sample is rotated with 100 mL of buffered extraction fluid at 37 °C for one hour. The supernatant is separated from the sample by filtration and analyzed for lead by an appropriate analytical method (e.g., Method 6010 and Method 6020).

3.0 DEFINITIONS

3.1 Bioavailability (BA) – The fraction of an ingested dose (i.e., *in vivo*) that crosses the gastrointestinal epithelium and becomes available for distribution to internal target tissues and organs.

3.2 Absolute bioavailability – Bioavailability expressed as a fraction (or percentage) of a dose.

3.3 Relative bioavailability (RBA) – The ratio of the bioavailability of a metal in one exposure context (i.e., physical chemical matrix or physical chemical form of the metal) to that in another exposure context. For this method, RBA is defined as the ratio of bioavailability of lead in soil to lead in water.

3.4 Bioaccessibility – An *in vitro* measure of the physiological solubility of the metal that may be available for absorption into the body.

3.5 Batch – A group of analytical and control/QC samples that are extracted simultaneously and is limited to 20 environmental samples in addition to the batch QC samples.

3.6 Phosphate-amended soil – phosphate rich materials (e.g., fertilizers) applied to lead-contaminated soils

3.7 *In vitro* – outside the living body and in an artificial environment

3.8 *In vivo* – in the living body of an animal

3.9 *In vitro* bioaccessibility (IVBA) – the physiological solubility of the metal that may be available for absorption into the body

3.10 Refer to Chapter One, Chapter Three, and the manufacturer's instructions for definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences during sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents may be necessary. Refer to each method to be used for specific guidance on QC procedures and to Chapters Three and Four for general guidance on glassware cleaning. Also refer to Methods 9040, 9045, 6010, 6020, 6800, and other determinative methods to be used for information regarding potential interferences.

4.2 At present, it appears that the predictive relationship between IVBA and RBA is widely applicable, having been found to hold true for a wide range of different soil types and lead phases from a variety of different sites. However, the majority of the samples tested have been collected from mining and milling sites, and it is plausible that some forms of lead that do not occur at these types of sites might not follow the observed correlation. Thus, whenever a sample containing an unusual and/or untested lead phase is evaluated by the IVBA protocol, this sample should be identified as a potential source of uncertainty. In the future, as additional samples with a variety of new and different lead forms are tested by both *in vivo* and *in vitro* methods, the limits on applicability of the method will be more clearly defined. In addition, excess phosphate in the sample medium may result in interference (i.e., the assay is not suited to phosphate-amended soils).

5.0 SAFETY

This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during the method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

This section does not list common laboratory glassware (e.g., beakers and flasks) that might be used.

This method recommends the use of a water bath (Section 6.1) or an incubated air chamber (Section 6.2).

6.1 Water Bath

If the water bath option is used, the specific extraction device is an electric motor (the same motor as is used in the toxicity characteristic leaching procedure (TCLP, Method 1311))

driven flywheel, which drives a rotating block situated inside a temperature-controlled water bath (See Figure 1). The extraction device must be capable of holding a capped 125-mL wide-mouth high density polyethylene (HDPE) bottle. The water bath should be filled such that the extraction bottles are completely immersed. Temperature in the water bath should be maintained at 37±2 °C using an immersion circulator heater, and the water bath temperature should be monitored and recorded. The electric motor must be capable of 30±2 rpm.

6.2 Incubated Air Chamber

If the air incubator option is used, the specific extraction device will rotate the extraction bottles within an incubated air chamber. It must be capable of rotating at 30±2 rotations per minute (rpm) and is designed to hold capped 125-mL wide-mouth HDPE bottles (see Figure 2 for an example of an extraction device in an incubated air chamber). The incubator must be capable of maintaining 37±2 °C. The temperature inside of the incubator should be monitored and recorded. Reference 17 presents results of a study comparing the use of a water bath with the use of an incubated air chamber for performing this method.

6.3 HDPE bottles, 125 mL in size, equipped with airtight screw-cap seals should be used. Care should be taken to ensure that the bottles do not leak and to minimize contamination during the extraction procedure.

6.4 Automated temperature compensation (ATC) pH electrode – used for measuring the pH of the extraction fluid both prior to and after the experiment

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals, at a minimum, should be used in all tests. Unless otherwise indicated, all reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS), where such specifications are available. Other grades may be used, provided the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Reagent water must be interference free. All references to water in this method refer to reagent water, unless otherwise specified.

7.3 Cleanliness of all materials used to prepare and/or store the extraction fluid and buffer is essential. All glassware and equipment used to prepare standards and reagents shall be properly cleaned, acid washed, and triple-rinsed with deionized water prior to use.

7.4 Extraction fluid – 0.4 M glycine (free base, reagent-grade glycine in deionized water), adjusted to a pH of 1.50±0.05 at 37 °C using trace metal-grade concentrated hydrochloric acid (HCl).

7.4.1 Prepare 2 liters (L) of extraction fluid in a volumetric flask (Class A) using American Society for Testing and Materials (ASTM) Type II deionized (DI) water. Add 60.06 grams of glycine (free base) to a flask containing 1.9 L of deionized water. Solution can be transferred to a wide-mouth HDPE bottle for ease of handling. Place the HDPE bottle containing the extraction fluid in a water bath at 37 °C and heat until the extraction fluid reaches 37 °C. Standardize the pH meter using an ATC pH electrode at 37 °C or pH buffers maintained at 37 °C in the water bath. Add trace metal-grade concentrated HCl (12.1 N) until the solution pH reaches 1.50±0.05. Bring the solution to a final volume of 2 L (0.4 M glycine).

7.4.2 If the extraction fluid is prepared in advance of the extraction, the extraction fluid must be heated to 37 °C and the pH shall be adjusted to 1.5 using trace metal grade concentrated HCl prior to conducting the extraction batch.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Sample collection, preservation and storage requirements may vary by EPA program and may be specified in a regulation or project planning document that requires compliance monitoring for a given contaminant. Where such requirements are specified in the regulation, those requirements must be followed. In the absence of specific regulatory requirements, see Chapters Three and Four in SW-846 as guidance in determining the sample collection, preservation and storage requirements.

Once the samples are prepared as described in Section 11.1, no preservatives or special storage conditions are required.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for guidance on QA and QC protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and Chapter One criteria; technique-specific QC criteria take precedence over Chapter One criteria. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a quality assurance project plan (QAPP) or a sampling and analysis plan (SAP), which translates project objectives and specifications into directions for those who will implement the project and assess the results.

Each laboratory should maintain a formal QA program. The laboratory should also maintain records to document the quality of the data generated. Development of in-house QC limits for each method is encouraged. Use of instrument-specific QC limits is encouraged, provided such limits will generate data appropriate for use in the intended application. All data sheets and QC data should be maintained for reference or inspection. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulatory community in making judgments necessary to generate results that meet the DQOs for the intended application.

9.2 Initial demonstration of proficiency (IDP)

Each laboratory must demonstrate initial proficiency by generating data of acceptable precision and bias for target analytes in a clean matrix. It is recommended that the laboratory repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation and/or procedures are made.

9.3 Reagent blank – Unprocessed (not run through the extraction procedure) extraction fluid should be analyzed for each new batch of extraction fluid. The reagent blank is considered within control limits if its result is less than the lower limit of quantitation (LLOQ). The corrective action for a blank hit above LLOQ should include preparing a new batch of extraction fluid and reprocessing any samples that were prepared with the failing reagent fluid.

9.4 Method blank – Extraction fluid only (i.e., no test soil) is carried through all steps of the method at a frequency of 1 in 20 samples (minimum of 1 per batch). The method blank is considered within control limits if its result is less than the LLOQ. The corrective action for a

recovery above the LLOQ should include making a new extraction fluid and reprocessing any samples that were prepared with the failing method blank.

9.5 Laboratory Control Sample (LCS) – A LCS consisting of a spiked blank may be run once per batch (minimum 1 in 20 samples). The LCS may be spiked with the same source as the calibration standards and needs to be carried through all steps of the rotation procedure. The control limits are 85-115% recovery. The corrective action for outliers should include an analyst review that all dilutions and spike concentrations were performed correctly. If no error is found, either re-extract the samples or flag and narrate the defect and possible bias in the data.

9.6 Matrix Spike (MS) – A MS should be run once per batch (minimum 1 in 20 samples). The MS should be prepared after extraction and filtration of the supernatant. The control limits are 75-125% recovery. The corrective action for outliers should include an analyst review that all dilutions and spike concentrations were performed correctly. If no error is found, either re-extract the samples or flag and narrate the defect and possible bias in the data.

9.7 Duplicate sample – A duplicate sample should be run once per batch (minimum 1 in 20 samples) and carried through all steps of the method. The relative percent difference (RPD) should be less than 20%. The corrective action for outliers should include either re-extraction of the samples or flagging the data.

9.8 Control soil – Any one of the following National Institute of Standards and Testing (NIST) standard reference materials (SRMs) may be used as a control soil: 2710a or 2711a (Montana soil). The reference material shall be carried through all steps of the method and analyzed at a frequency of 1 in 20 samples (minimum of 1 per batch). The IVBA is calculated using the equation in Sec. 12.3.1.

9.8.1 NIST SRM 2710a: Analysis of the NIST SRM 2710a standard should yield a mean IVBA result of 67.5% (acceptable IVBA range 60.7-74.2%). For the lead concentration (Pb_{soil}) in the SRM, the median lead concentration presented in the Addendum to the NIST certificate for leachable concentrations determined using Method 3050 (5,100 mg/kg) should be used.

9.8.2 NIST SRM 2711a: The NIST SRM 2711a should yield a mean IVBA result of 85.7% (acceptable IVBA range 75.2-96.2%). For the lead concentration (Pb_{soil}) in the SRM, the median lead concentration presented in the Addendum to the NIST certificate for leachable concentrations determined using Method 3050 (1,300 mg/kg) should be used.

9.8.3 NIST SRMs 2710a and 2711a are primary standard reference materials and are an integral part of the method quality control protocol. If NIST SRMs 2710a and 2711a are not available for purchase through the NIST website, check the following EPA website at: http://www.epa.gov/superfund/bioavailability/trw.htm or send an email to the EPA at bahelp@epa.gov to inquire about alternative SRMs.

9.9 Lower limit of quantitation check standard

9.9.1 The laboratory should establish the LLOQ as the lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve. The LLOQ should be verified by the analysis of at least seven replicate samples, which are spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases, the mean recovery should be ±35% of the true value and the RSD should be ≤20%. In-house

limits may be calculated when sufficient data points exist. The monitoring of recovery data for the LLOQ check standard over time is useful for assessing precision and bias. Refer to a scientifically valid and published method (such as Chapter 9 of *Quality Assurance of Chemical Measurements* (Taylor 1987)) or the Report of the Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs (http://water.epa.gov/scitech/methods/cwa/det/index.cfm) for calculating precision and bias for LLOQ.

9.9.2 Ongoing LLOQ verification, at a minimum, is carried out on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix (free of target compounds). Optimally, the LLOQ should be less than or equal to the desired regulatory action levels based on the stated project-specific requirements.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Prior to measurement of extraction fluid pH, the pH meter should be calibrated using a minimum of two points that bracket the expected pH (1.5) of the samples and are approximately two pH units or more apart. Repeat adjustments on successive portions of the two buffer solutions until readings are within 0.05 pH units of the buffer solution value as indicated in SW-846 method 9045D. The pH meter should be calibrated and checked with a standard that is of a different source from the buffers used to calibrate and within the calibration range (e.g., pH = 2) in accordance with the manufacturer's instructions.

10.2 Thermometers capable of measuring 37±2 °C are needed.

10.3 The analytical balance should be calibrated daily in accordance with the manufacturer's instructions.

10.4 Pipettes should be calibrated in accordance with the manufacturer's instructions and the laboratory QA plan.

11.0 PROCEDURE

11.1 All test soils should be prepared by drying (<40 °C) and sieving the sample as received to <250 µm. Milling should NOT be employed to achieve the desired particle size. The <250 µm size fraction is used because this particle size is representative of that which adheres to children's hands. Stainless steel sieves are recommended. Samples should be thoroughly mixed, prior to use, to ensure homogenization. The mixing and aliquoting of samples using a riffle splitter is recommended. The use of clean HDPE storage bottles is recommended.

11.2 The extraction fluid for this procedure is 0.4 M glycine (free base, reagent-grade glycine in deionized water), adjusted to a pH of 1.50±0.05 at 37±2 °C using trace-metal grade concentrated hydrochloric acid (HCl). The extraction fluid should be pre-heated to 37±2 °C. See Sec. 7.5 for extraction fluid preparation details.

11.3 Pre-heat the TCLP extractor water bath or incubator (See Sec. 6.0) to 37 °C. Record the temperature at the beginning and end of each extraction batch.

11.4 Soil samples should be thoroughly mixed immediately prior to subsampling for extraction to ensure homogenization (i.e., rotate sample bottles using X, Y, Z motion).

11.5 The extraction procedure begins by placing 1.00±0.05 g of sieved test material (<250 µm) into a 125-mL wide-mouth HDPE bottle. Record the weight of the soil to the nearest 0.0001 g. Care should be taken to ensure that static electricity does not cause soil particles to adhere to the lip or outside threads of the bottle. If necessary, an antistatic brush should be used to eliminate static electricity prior to adding the test substrate.

11.6 Measure 100±0.5 mL of the 37±2 °C buffered extraction fluid (0.4 M glycine, pH 1.5), using a graduated cylinder or automated dispenser and transfer the extraction fluid to the 125-mL wide-mouth HDPE bottle.

11.7 The bottle should be tightly sealed and then shaken or inverted to ensure that there is no leakage and that no soil is caked on the bottom of the bottle.

NOTE: Care should be taken to prevent contamination of the samples during rotation (e.g., getting bath water in the threads around the cap and possibly into the sample when the cap is removed). Precautions that laboratories may consider include but are not limited to: the type of bottle that is used, sealing the samples in plastic freezer bags with air expelled before installing in the water bath extractor, and/or sealing the bottles with tape or Parafilm®.

11.8 Fill the extractor (TCLP extractor or rotating extractor inside of a pre-heated incubator, see Sec. 6.0 for details) with 125-mL bottles containing test materials or QC samples (see Sec. 7.0). Record start time of rotation.

11.9 Samples are extracted by rotating the samples at 30±2 rpm for one hour.

11.10 After one hour, the bottles should be removed from the rotator, dried, and placed upright on the bench top to allow the soil to settle to the bottom.

11.11 A 40-mL sample of supernatant fluid is then removed directly from the extraction bottle into a disposable syringe. After withdrawal of the sample into the syringe, a Luer lock attachment (equipped with a 0.45-µm cellulose acetate disk filter (25-mm diameter)) is attached, and the sample is filtered through the attached disk filter to remove any particulate matter into a clean (e.g., acid-washed or pre-cleaned) polypropylene centrifuge tube or other appropriate sample vial for analysis.

11.12 Record the time that the extract is filtered (i.e., extraction is stopped). If the total time elapsed for the extraction and filtration process exceeds 90 minutes, the test must be repeated (i.e., Steps 11.1-11.11).

11.13 Measure and record the temperature and pH of fluid remaining in the extraction bottle. If the fluid pH is not within ±0.5 pH units of the starting pH, the test must be discarded and the sample reanalyzed.

NOTE: In some cases (mainly slag soils), the test material can increase the pH of the extraction buffer and this could influence the results of the bioaccessibility measurement. To guard against this, the pH of the fluid should be measured at the end of the extraction step (just after a sample was withdrawn for filtration and analysis). If the pH is not within 0.5 pH units of the starting pH (1.5), the sample should be re-extracted. If the second test also results in an increase in pH of >0.5 units, it is reasonable to conclude that the test material is buffering the solution. In these cases, the test should be repeated using

manual pH adjustment during the extraction process, stopping the extraction at 5, 10, 15, and 30 minutes and manually adjusting the pH down to pH 1.5 at each interval by drop-wise addition of trace-metal grade HCl.

11.14 Store filtered sample(s) in a refrigerator at 4±2 °C until they are analyzed. This filtered sample of extraction fluid is then analyzed for lead by an appropriate method (see Sec. 2.0 for examples of appropriate methods).

NOTE: In some cases high dissolved solids in the extracts may cause nebulizer performance issues by inductively coupled plasma-optical emission spectrometry (ICP-OES) or inductively coupled plasma-mass spectrometry (ICP-MS). If this is encountered, dilution of the extracts tenfold is recommended before analysis. Correct for any dilutions in the calculations. Alternately, a high solids nebulizer may be useful. Graphite furnace atomic absorption spectrophotometry (GFAA) should be avoided due to the high levels of HCl in the extracts.

Note: In some cases, the amount of lead present in the sample will begin to saturate the extraction fluid, and the extraction response will cease to be linear. If the concentration of lead in the extract exceeds approximately 500 mg/L (depending on the sample matrix and mineralogy), this upper limit may have been reached. It is not recommended to analyze IVBA for soils exceeding a total lead concentration of 50,000 mg/kg in order to avoid saturation of the extraction fluid and because risk management decisions are not likely to be improved by analyzing IVBA for soil with concentrations of lead above this level. Reference 19 can be consulted for more information on how different liquid to solid ratios impact the bioaccessibility of metals in soils.

11.15 A check list of minimum data recording requirements is provided in Sec. 17.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 If the IVBA factor is to be determined, a split of each solid material (<250 µm) that has been subjected to this extraction procedure should be analyzed for total lead concentration using analytical procedures taken from SW-846 or a non-destructive method such as Instrumental Neutron Activation Analysis. If SW-846 methods are used, the solid material should be acid digested according to an appropriate preparation method (e.g., Method 3050 or Method 3051). The digestate should be analyzed for lead concentration by an appropriate analytical method.

NOTE: Since this method may be applied to samples containing high amounts of lead, the analyst should read section 8.4 of Method 3050 in case linear range or digestion capacity are exceeded for high level samples.

12.2 If dilutions were performed, apply the appropriate corrections to the sample values.

12.3 *In vitro* bioaccessibility (IVBA)

12.3.1 The IVBA is calculated and expressed on a percentage basis using the following equation:

Where: Pb_{ext} = *in vitro* extractable lead in the *in vitro* extract (mg/L)
V_{ext} = extraction solution volume (L)
Pb_{soil} = lead concentration in the soil sample being assayed (mg/kg)
$Soil_{mass}$ = mass of soil sample being assayed (kg)

12.3.2 In order for an *in vitro* bioaccessibility test system to be useful in predicting the *in vivo* RBA of a test material, it is necessary to empirically establish that a strong correlation exists between the *in vivo* and the *in vitro* results across many different samples (see Reference 10). Due to the measurement error in RBA, as well as in IVBA, a linear regression calibration fit was used to minimize the error in both the RBA and IVBA approach. There was no significant difference in fit observed, so the results of the weighted linear regression were selected for simplicity. This decision may be revisited as more data become available. Based on the available data, the currently preferred calibration model is:

where RBA and IVBA are expressed as fractions, not as percentages. It is important to recognize that the use of this equation to calculate RBA from a given IVBA measurement will yield the "typical" RBA value expected for a test material with that IVBA, and the true RBA may be somewhat different (either higher or lower).

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. Performance data must not be used as absolute QC acceptance criteria for purposes of laboratory QC or accreditation.

13.2 Refer to the appropriate determinative method for performance data examples and guidance.

13.3 Information on the recent round-robin study used to develop the new lead IVBA means (calculation for percent IVBA is located in Sec. 12.3) for NIST 2710a and 2711a are provided in Reference 9. This data is provided for guidance purposes only.

13.4 Reference 17 presents results of a study comparing the use of a water bath with the use of an incubated air chamber for performing this method. This data is provided for guidance purposes only.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the EPA recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction,* a free publication available from the ACS, Committee on Chemical Safety, http://portal.acs.org/portal/fileFetch/C/WPCP_012290/pdf/WPCP_012290.pdf.

15.0 WASTE MANAGEMENT

The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Laboratories are urged to protect air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the web address listed in Sec. 14.2.

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17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables and figures referenced by this method.

TABLE 1
LABORATORY RESULTS AND THE PREDICTION AND
CONFIDENCE INTERVALS FOR NIST 2710A

NIST 2710a Analyte: Lead Units: mg/Kg							
Laboratory	A	B	C	D	E	F	G
Extraction Type	Water	Water	Water	Water	Water	Air	Air
Rep 1	3290	3520	3320	3567.5	3652.5	3372	3430
Rep 2	3270	3470	3300	3592.6	3623.4	3314	3370
Rep 3	3290	3483	3360	3495.6	3663.2	3321	3420
Rep 4	3300	3479	3330	3536.2	3632.6	3347	3430
Rep 5	3290	3538	3370	3617.0	3605.6	3348	3460
Average	3288.0	3498.0	3336.0	3561.8	3635.5	3340.4	3422.0
Std Dev	10.95	29.39	28.81	47.61	22.94	23.31	32.71
RSD	0.33	0.84	0.86	1.34	0.63	0.70	0.96

Pooled n=35	
Average	3440.23
Std Dev	124.58
RSD	3.62

Extracted Pb 99 - Percentile Prediction Interval (mg/Kg)		
99 low	Average	99 high
3095.56	3440.23	3784.91
10.02% = ± 99 prediction interval in percent		

Lead IVBA 99 - Percentile Prediction Interval		
99 low	Average	99 high
60.70	67.46	74.21
NIST 2710a Digestion EPA Method 3050 median result from the NIST certificate is 5100 mg/Kg		
IVBA = 67.46 or 67.5%	SD = 2.44	RSD = 3.62

Confidence Interval of the Mean		
3440.23 = Mean	21.05798 = SD of the Mean	0.61 = RSD of the Mean
99 low	Average	99 high
3382.79	3440.23	3497.68
1.67 % = ± 99 percentile of the confidence interval of the mean		

Std Dev = Standard Deviation
RSD = Relative Standard Deviation
CI = Confidence Interval

Source: Shaw 2011

TABLE 2
SRM 2710A BATCH QC SAMPLE RESULTS, LEAD

Laboratory	A	B	C	D	E	F	G	Mean
Extraction Type	Water	Water	Water	Water	Water	Air	Air	
Reagent Blank <25 ug/L	<30	<5	<40	<0.95	1.98	2.67	9.6	na
Bottle Blank ug/L <50 ug/L	<30	<5	<40	<0.95	1.86	NA	5.1	na
Blank Spike Percent Recovery (85-115%)	96.1	98.6	96.3	99.0	100.0	97.0	98.0	97.9
Control Soil SRM 2711 mg/Kg (nominal =928.4 mg/Kg)	865	953	910	977.8	1007.2	906.6	953	938.9
IVBA Control Soil SRM 2711 mg/Kg IVBA = 84.4 (%)	78.6%	86.6%	82.7%	88.9%	91.6%	82.4%	86.6%	85.4%
IVBA Control Soil SRM 2711 Percent Recovery (%)	93.2%	102.6%	98.0%	105.3%	108.5%	97.7%	102.6%	101.1%

na = not applicable

Source: Shaw 2011

TABLE 3
NIST 2710A ROUND ROBIN RESULTS ANALYSIS OF VARIANCE

ANOVA: Single Factor (Lead)
Note: alpha at 0.05 (95 percentile)

Groups	Count	Sum	Average	Variance
Laboratory A	5	16440	3288	120
Laboratory B	5	17490	3498	864
Laboratory C	5	16680	3336	830
Laboratory D	5	17809	3562	2266
Laboratory E	5	18177	3635	526
Laboratory F	5	16702	3340	543
Laboratory G	5	17110	3422	1070

ANOVA

Source of Variation	SS	df	MS	F	P-value	F crit
Inter-laboratory	502813.7789	6	83802.29648	94.31778649	2.93938E-17	2.445259395
Intra-laboratory	24878.28	2	888.51			
Total	527692.0589	34				

SS = Sum of Squares
df = Degrees of Freedom
MS = Mean Square
F = F Value Calculated
F Crit = Critical Value of F
P-value = Probability Value

Source: Shaw 2011

TABLE 4
NIST SRM 2710A RESULTS, AIR VERSUS WATER TEMPERATURE CONTROL MEDIUM, T-TEST

NIST 2710a Analyte: Lead Units: mg/Kg									
Extraction Type	WATER							AIR	
Laboratory	A	B	C	D	E			F	G
Rep 1	3290	3520	3320	3567.5	3652.5		Rep 1	3372	3430
Rep 2	3270	3470	3300	3592.6	3623.4		Rep 2	3314	3370
Rep 3	3290	3483	3360	3495.6	3663.2		Rep 3	3321	3420
Rep 4	3300	3479	3330	3536.2	3632.6		Rep 4	3347	3430
Rep 5	3290	3538	3370	3617	3605.6		Rep 5	3348	3460
Average	3288.0	3498.0	3336.0	3561.8	3635.5		Average	3340.4	3422.0
Std Dev	10.95	29.39	28.81	47.61	22.94		Std Dev	23.31	32.71
RSD	0.33	0.84	0.86	1.34	0.63		RSD	0.70	0.96

	WATER	PERCENT DIFFERENCE	AIR
	n=25	2.41%	n=10
AVG	3463.9	AVG	3381.2
Std Dev	137.8	Std Dev	50.7
RSD	4.0	RSD	1.5

Excel t-Test: Two-Sample Assuming Equal Variances
alpha = 0.05

	WATER	AIR
Mean	3463.85	3381.2
Variance	18991.74177	2566.622222
Observations	25	10
Pooled Variance	14512.16371	
Hypothesized Mean Difference	0	
df	33	
t Stat	1.833590061	
P(T ≤ t) two-tail	0.075747815	
t Critical two-tail	2.034515287	

t-Stat = t-statistic

t crit = t critical value

P(T ≤ t) two tail = if the value is less than 0.05 indicates a 95% probability that the means of the two groups do not come from the same population

Source: Shaw 2011

TABLE 5
LABORATORY RESULTS AND THE PREDICTION AND
CONFIDENCE INTERVALS FOR NIST 2711A

NIST SRM 2711a Analyte: Lead Units: mg/Kg							
Laboratory	A	B	C	D	E	F	G
Extraction Type	Water	Water	Water	Water	Water	Air	Air
Rep 1	1040	1145	1080	1138.3	1181.7	1099	1130
Rep 2	1030	1147	1100	1121.3	1194.2	1057	1130
Rep 3	1040	1122	1080	1155.1	1177.6	1089	1130
Rep 4	1030	1157	1080	1150.8	1182.2	1086	1120
Rep 5	1030	1165	1060	1151.1	1190.8	1082	1130
Average	1034.0	1147.2	1080.0	1143.3	1185.3	1082.6	1128.0
Std Dev	5.48	16.22	14.14	13.83	6.92	15.63	4.47
RSD	0.53	1.41	1.31	1.21	0.58	1.44	0.40

Pooled	n=35
Average	1114.4
Std Dev	49.4
RSD	4.4

Extracted Pb 99 – Percentile Prediction Interval (mg/Kg)		
99 low	Average	99 high
979.64	1114.35	1249.05
12.09 = ± 99 percentile prediction interval in percent		

IVBA 99-Percentile Prediction Interval		
99 low	Average	99 high
75.21	85.72	96.23
NIST 2711a Digestion EPA Method 3050 the median result from the NIST certificate of analysis is 1300 mg/Kg		
so IVBA =85.72 or 85.7% SD= 3.80 RSD = 4.43		

Confidence Interval of the Mean at 99 percentile		
1114.35 = Mean	8.346 = SD of the Mean	0.749 = RSD of the Mean
99 low	Average	99 high
1091.58	1114.35	1137.11
2.04% = ± 99 percentile confidence interval of the mean		

Std Dev = Standard Deviation
RSD = Relative Standard Deviation
CI = Confidence Interval

Source: Shaw 2011

TABLE 6
SRM 2711A BATCH QC SAMPLE RESULTS, LEAD

Laboratory	A	B	C	D	E	F	G	Mean
Extraction Type	Water	Water	Water	Water	Water	Air	Air	
Reagent Blank <25 ug/L	<30	<5	<40	<0.95	1.7	0.55	11.4	na
Bottle Blank ug/L <50 ug/L	<30	<5	<40	<0.95	1.42	nr	4.6	na
Blank Spike Percent Recovery (85-115%)	95.7%	96.6%	95.7%	95%	98.6%	98%	98%	96.8%
Control Soil SRM 2711 mg/Kg (nominal =928.4 mg/Kg)	861.1	967	900	958.8	1014	921.7	958	940.1
IVBA Control Soil SRM 2711 mg/Kg IVBA = 84.4 (%)	78.3%	87.9%	81.8%	87.2%	92.2%	83.8%	87.1%	85.5%
IVBA Control Soil SRM 2711 Percent Recovery (%)	92.8%	104.2%	96.9%	103.3%	109.2%	99.3%	103.2%	101.3%

nr = not reported na = not applicable

Source: Shaw 2011

TABLE 7
NIST 2711A ROUND ROBIN RESULTS ANALYSIS OF VARIANCE

Anova: Single Factor (Lead)
note alpha at 0.05 (95 percentile)

Groups	Count	Sum	Average	Variance
Laboratory A	5	5170	1034	30
Laboratory B	5	5736	1147	263
Laboratory C	5	5400	1080	200
Laboratory D	5	5716.6	1143	191
Laboratory E	5	5926.5	1185	47.8
Laboratory F	5	5413	1083	244
Laboratory G	5	5640	1128	20

ANOVA

Source of Variation	SS	df	MS	F	P-value	F crit
Inter-laboratory	78913.57886	6	13152.26314	92.37418704	3.87283E-17	2.445259395
Intra-laboratory	3986.648	28	142.3802857			
Total	82900.22686	34				

SS = Sum of Squares
df = Degrees of Freedom
MS = Mean Square
F = F Value Calculated
F Crit = Critical Value of F
P-value = Probability Value

Source: Shaw 2011

TABLE 8
NIST SRM 2711A RESULTS, AIR VERSUS WATER TEMPERATURE
CONTROL MEDIUM, T-TEST

NIST 2711a Analyte: Lead Units: mg/Kg								
Extraction Type>	WATER						AIR	
Laboratory>	A	B	C	D	E		F	G
Rep 1	1040	1145	1080	1138.3	1181.7		1099	1130
Rep 2	1030	1147	1100	1121.3	1194.2		1057	1130
Rep 3	1040	1122	1080	1155.1	1177.6		1089	1130
Rep 4	1030	1157	1080	1150.8	1182.2		1086	1120
Rep 5	1030	1165	1060	1151.1	1190.8		1082	1130
AVG	1034.0	1147.2	1080.0	1143.3	1185.3		1082.6	1128.0
Std Dev	5.48	16.22	14.14	13.83	6.92		15.63	4.47
RSD	0.53	1.41	1.31	1.21	0.58		1.44	0.40

	WATER	PERCENT DIFFERENCE		AIR
	n=25	1.14		n=10
AVG	1117.96		AVG	1105.30
Std Dev	56.10		Std Dev	26.27
RSD	5.02		RSD	2.38

Excel t-Test: Two-Sample Assuming Equal Variances
alpha = 0.05

	Water	Air
Mean	1117.964	1105.3
Variance	3147.690733	690.0111111
Observations	25	10
Pooled Variance	2477.414473	
Hypothesized Mean Difference	0	
df	33	
t Stat	0.679997865	
P(T ≤ t) two-tail	0.501248691	
t Critical two-tail	2.034515287	

t-Stat = t-statistic

t crit = t critical value

P(T ≤ t) two tail = if the value is less than 0.05 indicates a 95% probability that the means of the two groups do not come from the same population

Source: Shaw 2011

TABLE 9

ROUND ROBIN STUDY SRM IVBA RESULTS COMPARED TO PREVIOUS IVBA RESULTS

SRM	Mean IVBA	Standard Deviation	RSD	CV	N
2710 Previous Lot	75.5%	4.7	6.2	0.062	68
2711 Previous Lot	84.4%	4.7	5.5	0.055	66
2711 This Study	85.4%	4.3	5.0	0.050	14
2710a	67.5%	2.4	3.6	0.036	35
2711a	85.7%	3.8	4.4	0.044	35

Source: Shaw 2011

TABLE 10

NIST SRMS 2710A AND 2711A 99 PERCENTILE ROUNDED VALUES

SRM	Low 99	Average	High 99
SRM 2710a (mg/Kg)	3100	3440	3780
SRM 2710a IVBA	60.7	67.5	74.2
SRM 2711a (mg/Kg)	980	1110	1250
SRM 2711a IVBA	75.2	85.7	96.2

Source: Shaw 2011

FIGURE 1

EXAMPLE OF AN *IN VITRO* BIOACCESSIBILITY EXTRACTION APPARATUS WITH WATER BATH

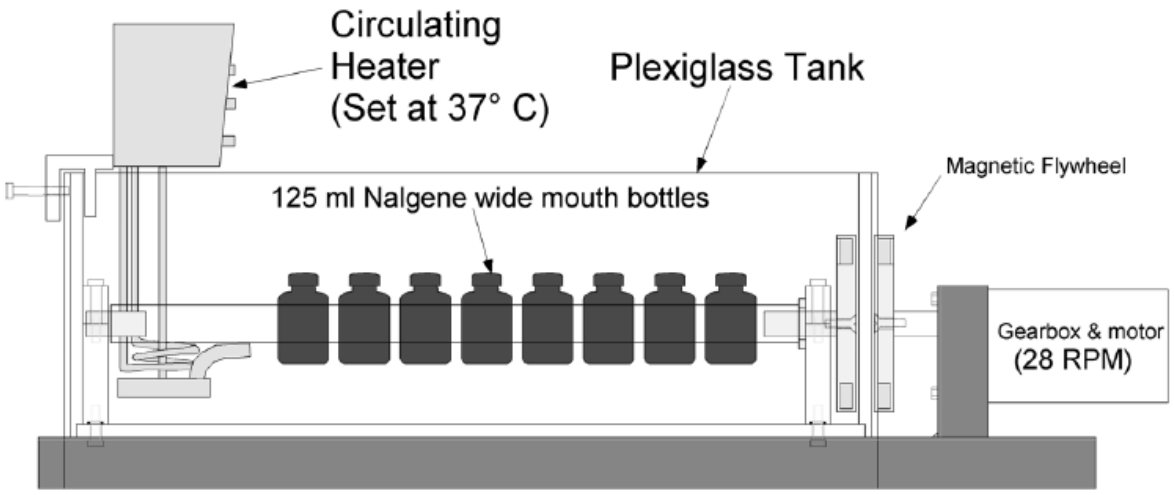
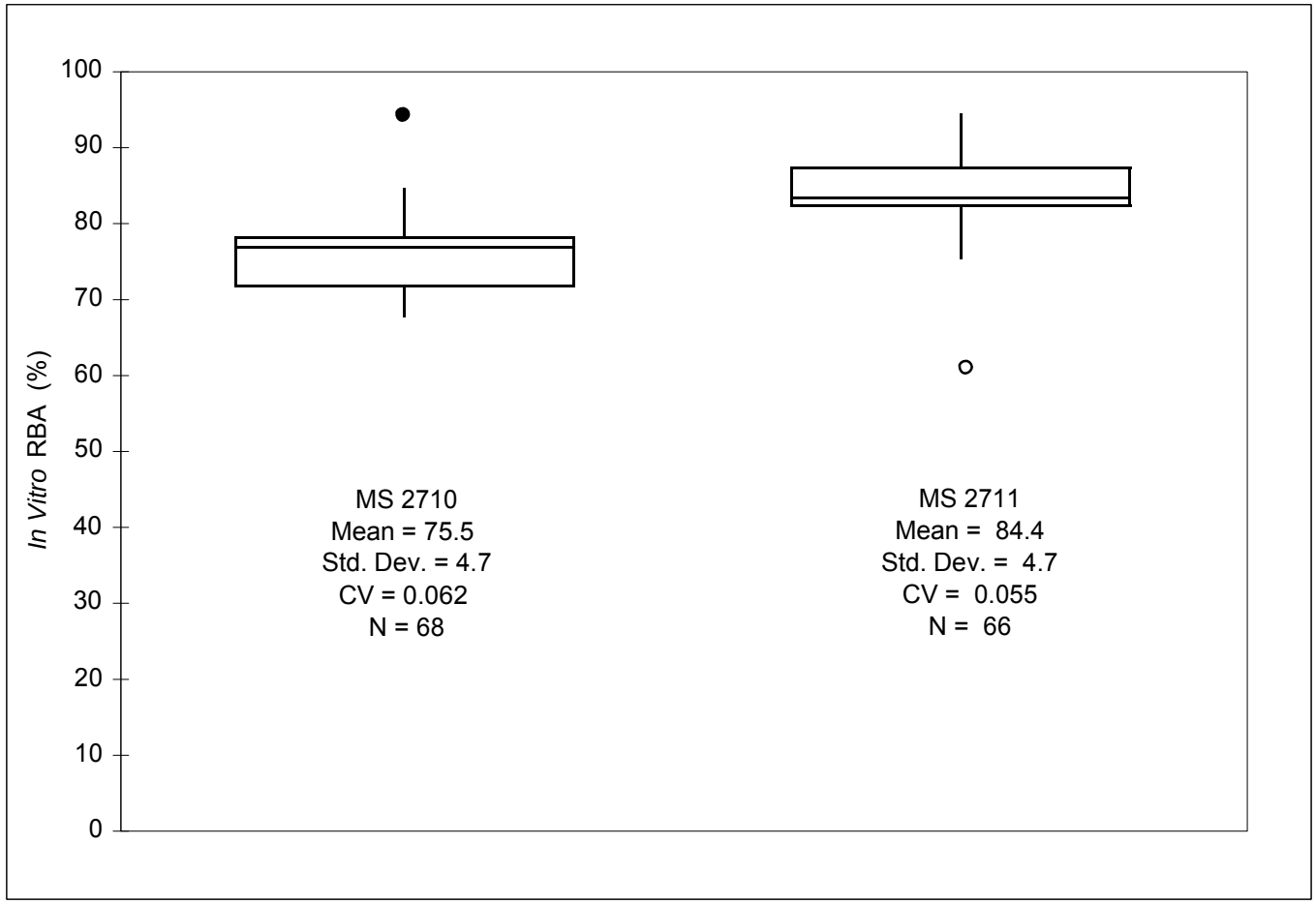


FIGURE 2

EXAMPLE OF AN *IN VITRO* BIOACCESSIBILITY EXTRACTION APPARATUS IN AN AIR INCUBATOR

FIGURE 3

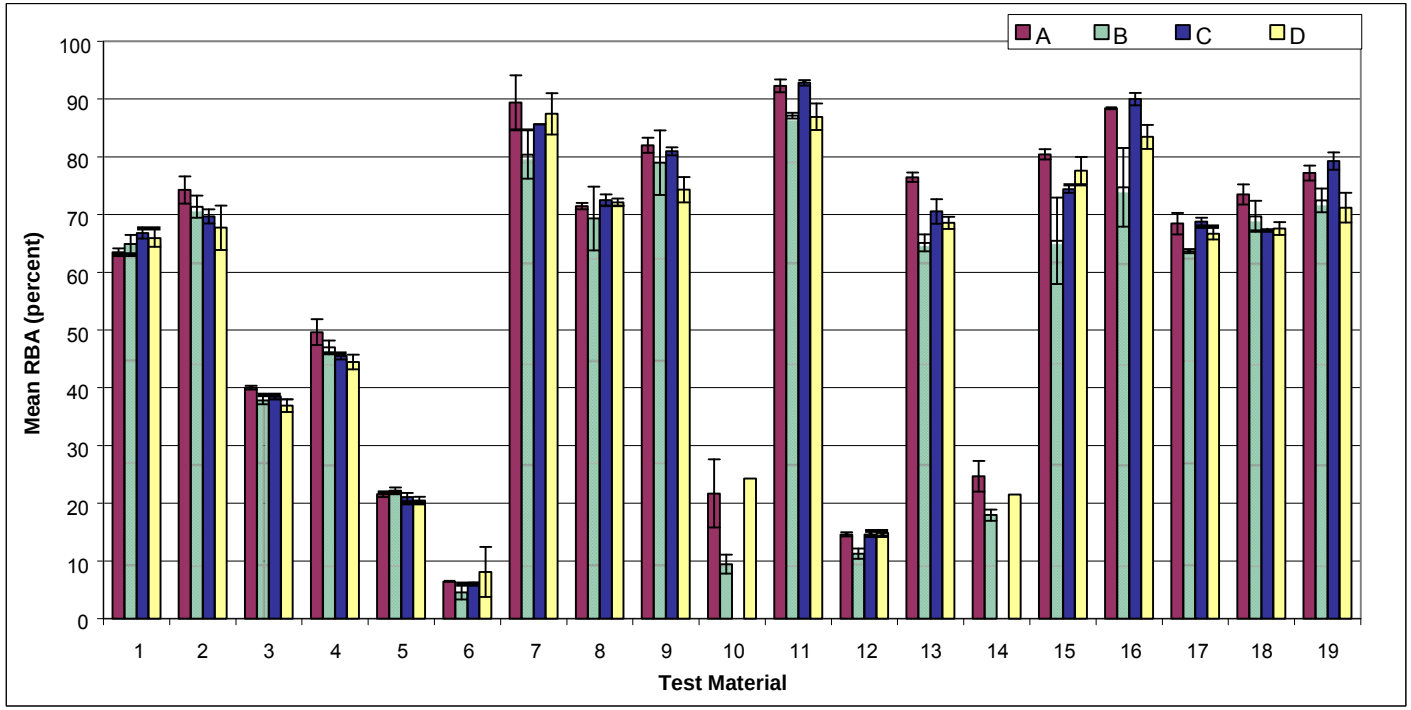
PRECISION OF *IN VITRO* BIOACCESSIBILITY MEASUREMENTS



Source: OSWER 2007b

FIGURE 4

REPRODUCIBILITY OF *IN VITRO* BIOACCESSIBILITY MEASUREMENTS

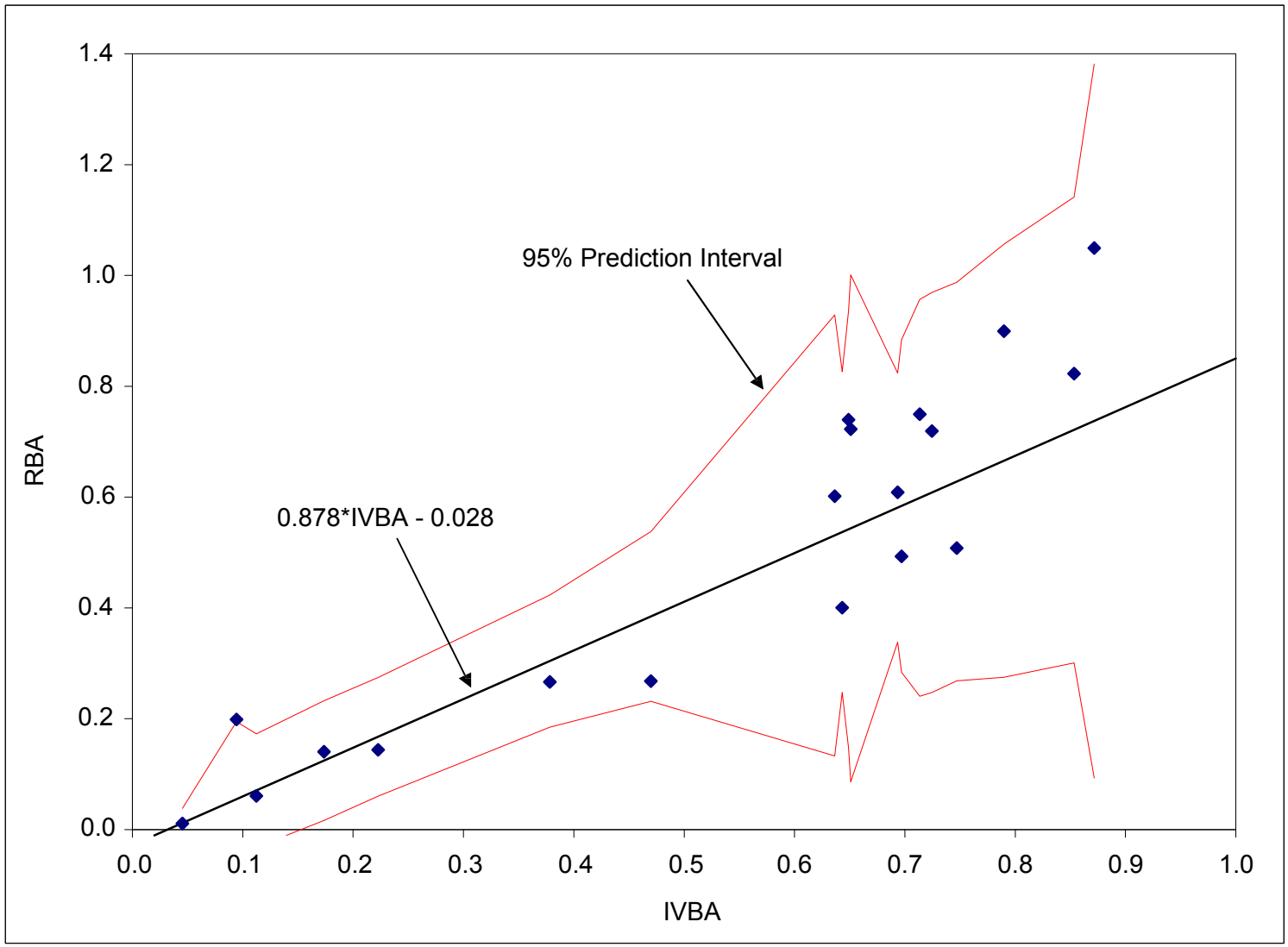


Test Materials

1 = Aspen Berm	8 = Jasper County High Lead Smelter	14 = Midvale Slag
2 = Aspen Residential	9 = Jasper County Low Lead Yard	15 = Murray Smelter Slag
3 = Bingham Creek Channel Soil	10 = California Gulch AV Slag	16 = Murray Smelter Soil
4 = Bingham Creek Residential	11 = California Gulch Fe/Mn PbO	17 = Palmerton Location 2
5 = Butte Soil	12 = California Gulch Oregon Gulch Tailings	18 = Palmerton Location 4
6 = Galena-enriched Soil	13 = California Gulch Phase I Residential Soil	19 = NIST Paint
7 = Jasper County High Lead Mill		

Source: OSWER 2007b

FIGURE 5
PREDICTION INTERVAL FOR RBA BASED ON MEASURED IVBA



Source: OSWER 2007b

FIGURE 6 EXAMPLE EXTRACTION RECORD

Date: **Sample ID:**
BATCH No:
Extraction Fluid ID: Glycine & HCl, pH 1.5; **SRM ID:**
Spike solution concentration: 10mg/L Pb
Lead Spiking Solution Vendor, Lot No. (X mL of standard added to X mL extraction solutions (100mL total volume) labeled as "spikes")

	Sample Preparation			Extraction					
Sample ID	**Bottle No.**	**Volume (mL)**	**Sample Mass (g)**	**Time (min)**	**Initial pH**	**Final pH**	**Start Temp (C)**	**End Temp (C)**	**Total Time (min)**
Acceptance Range		**100 ± 0.5**	**1.00 ± 0.01**	**60 ± 5**	**1.50 ± 0.05**	**1.50 ≥ 0.50**	**37 ± 2**	**37 ± 2**	**≤ 90**
Bottle Blank	1		NA						
Blank Spike	2		NA						
NIST SRM ID	3								
Sample ID	4								
Sample ID	5								
Sample ID	6								
Sample ID	7								
Sample ID	8								
Sample ID	9								
Sample ID	10								
Sample ID	11								
Sample ID	12								
reagent blank	13		NA	NA	NA	NA	NA	NA	NA

reagent blank is not extracted through in vitro process

FIGURE 7

GASTRIC EXTRACTION FLUID PREPARATION

	Sample Batch No: Date Prepared:				
Component	Lot ID	Fluid Preparation		Actual Quantity	Comments
		1 L	2 L		
Deionized Water	ASTM Type II	0.95 L (approx.)	1.90 L (approx.)		
Glycine	Sigma Lot No.	30.04±0.05g	60.08±0.05g		
HCL (12.1 N; Tr.metal)	Fisher Optima	(approx.)	(approx.)		
Final Volume	_____	1.0 L (class A)	2.0 L (class A)		
pH at 37C	_____	1.50±0.05	1.50±0.05		

FIGURE 8 EXAMPLE BATCH FORMAT AND IVBA CALCULATION

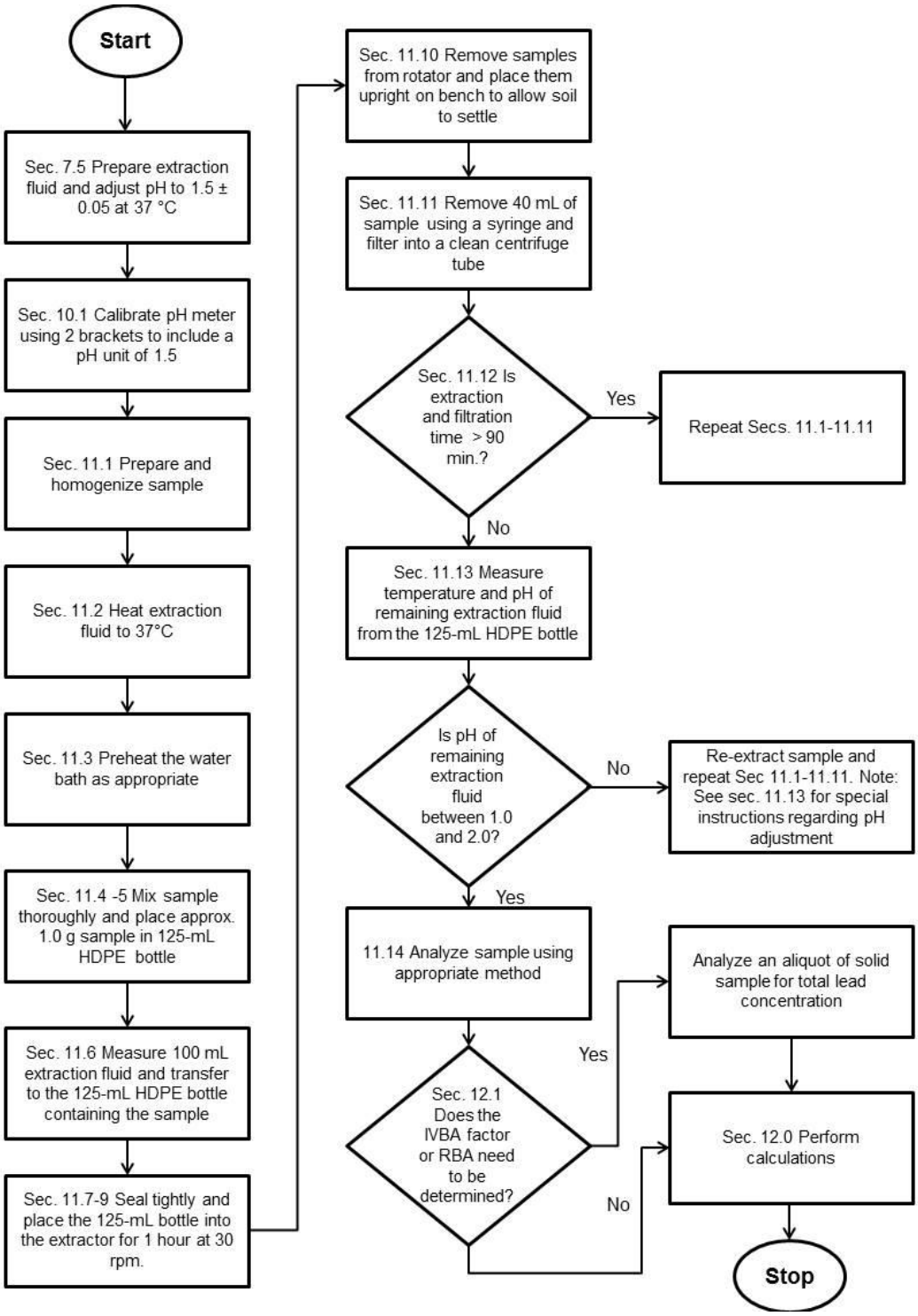
Date: **Sample ID:**
BATCH No:
Extraction Fluid ID: Glycine & HCl, pH 1.5; **SRM ID:**
Spike solution concentration: 10mg/L Pb
Lead Spiking Solution Vendor, Lot No. (X mL of standard added to X mL extraction solutions (100mL total volume) labeled as "spikes")

Batch #	Bottle No.	Type	Sample ID	Soil weight	Soil weight	Volume	Volume	ICP (Pb)	Soil [Pb]	% IVBA	Avg	S.D. of % IVBA
				grams	kg	(ml)	(L)	mg/L	(mg/kg)		% IVBA	
Insert No.	1	QC	Bottle blank	n/a	n/a	100	0.1		n/a	n/a		
	2	Blank spike	Blank + spike	n/a	n/a	100	0.1		n/a	n/a		
	3	Control soil	SRM 2710a	1.0019	0.00100	100	0.1	34.24	5100	**67**		
	4	Sample	Sample1 a	1.0016	0.00100	100	0.1	32.24	5100	**63**	**64.1**	**1.4**
	5	Sample	Sample1 b	1.0006	0.00100	100	0.1	33.24	5100	**65**		
	6	Matrix spike	Sample + spike	0.9985	0.00100	100	0.1					
	7	Sample	Sample2 a	1.0029	0.00100	100	0.1				Avg of dups	SD
	8	Sample	Sample2 b	1.0022	0.00100	100	0.1					
	9	Matrix spike	Sample + spike	1.0028	0.00100	100	0.1					
	10	Sample	Sample3 a	1.0004	0.00100	100	0.1				Avg of dups	SD
	11	Sample	Sample3 b	1.0029	0.00100	100	0.1					
	12	Matrix spike	Sample + spike	0.9972	0.00100	101	0.1		n/a	n/a		
	13	Reagent blank	unprocessed sample	n/a	n/a	100	0.1		n/a	n/a		

Example calculation: % IVBA = $\frac{\text{(Concentration in IVBA extract mg/L)(0.1 L)}}{\text{(Concentration in solid mg/kg)(weight of sample kg)}}$ *** 100**

METHOD 1340 FLOWCHART

IN VITRO BIOACCESSIBILITY ASSAY FOR LEAD IN SOIL



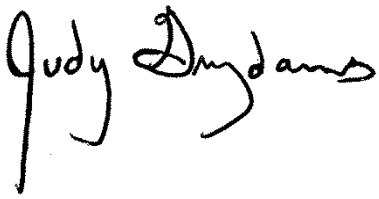
Corvallis ASL Standard Operating Procedure

STANDARD OPERATING PROCEDURE
FOR PHYSIOLOGICALLY BASED EXTRACTION PROCEDURE (PBEP)

METHOD BASED ON THE FOLLOWING SOURCE METHODS:

Preparatory Methods	**Analytical Methods**
In House	SW6010B (1996)
	SW6010C (2007)
	SW6020 (1994)
	SW6020A (1998)

APPROVED:



2/23/15

Section Leader — Date

STANDARD OPERATING PROCEDURE
FOR PHYSIOLOGICALLY BASED EXTRACTION PROCEDURE (PBEP)

1.0 SCOPE AND APPLICATION

This SOP describes the procedures used by CH2M HILL Applies Sciences Laboratory to determine the bio-accessibility of lead and/or arsenic for soils.

2.0 OVERVIEW OF THE ANALYTICAL PROCESS

Soil samples are dried, sieved, added to an extraction fluid, and extracted one hour at a temperature of 37ºC with constant agitation. After extraction the aqueous phase is separated from the solid phase and analyzed by ICP or ICPMS.

3.0 TARGET ANALYTES, REPORTING LIMITS, AND DETECTION LIMITS

All reporting limits, QC frequency, and QC acceptance criteria are subject to change on a client specific basis as requested by that client.

3.1 Target analytes are arsenic and lead.

4.0 INTERFERENCES

Not applicable.

5.0 SAFETY, WASTE MINIMIZATION, AND POLUTION PREVENTION

5.1 Laboratory wastes shall be separated and properly disposed complying with all federal, state, and local regulations. The wastes include collected solvent rinses, expired sample extracts, and disposable labware (or other item as applicable) used in the preparation of the samples. These wastes shall be handled according to ASL SOP HAZ01, Waste Disposal.

5.2 Analysts are encouraged to reduce the amount of solvent or disposable labware waste whenever possible. More information on this topic can be found in "Less is Better: Laboratory Chemical Management Waste Reduction" from the American Chemical Society.

5.3 Lab analysts shall wear lab coats, safety glasses, and surgical gloves when preparing and handling standards and field and lab samples

6.0 SAMPLE COLLECTION, STORAGE, HOLDING TIMES, AND PRESERVATION

Samples are transported on ice. Samples are stored at 4º C. Holding time for metal analyses is 6 months. Extracts are stored at room temperature.

7.0 APPARATUS AND MATERIALS

All purchasing of apparatus, materials, standards, gases, and reagents will be completed according to the SOP on Purchasing and Receipt of Standards and Reagents ASL SOP31. Support equipment and instrumentation utilized in this SOP and requiring metrological compliance either on an annual or quarterly basis can be found listed in the Metrology and Equipment Verification matrix.

7.1 Hot Water Bath (37 ± 2ºC)

7.2 Sieve (#60 / 250microns)
7.3 Analytical balance readable to 0.01-g
7.4 250mL bottle with air tight screw-cap seal or equivalent vessel that will not leak during extraction
7.5 pH meter
7.6 Centrifuge
7.7 Filtration Unit: Sterivex 0.45um filters

8.0 STANDARDS, GASES, AND REAGENTS

Any standard that is prepared in the laboratory shall be verified against current standards, or in the case of calibration standards against a second source calibration verification standard, prior to use. This verification shall be recorded in the standard logbook.

8.1 HCl – OPTIMA grade HCl
8.2 SBRC protocol Extraction Fluid: Add 60.06g glycine to 1.9L UPW. Place in hot water bath until temperature of fluid reaches 37ºC. Add concentrated HCl until pH reaches 1.50±0.05 (~60mL). Bring the volume to 2L using UPW. Suggested holding time is 2 days.
8.3 Bioaccessibility of Ingested Mine-Waste Lead Extraction Fluid: Add 50mg of pepsin, 0.50g of citrate, 0.50g of malate, 420uL of lactic acid, and 500uL of acetic acid to ~20mL of UPW. Homogenize and bring the volume to 40mL with UPW. Adjust the pH to 1.3 using HCl. Suggested holding time for the extraction fluid is 2 days.
8.4 PBET Extraction Fluid: Add 1.25g of pepsin, 0.50g of citrate, 0.50g of malate, 420uL of lactic acid, and 500uL of acetic acid to ~500mL UPW. Homogenize and bring the volume to 1L with UPW. Adjust the pH to 1.3 using HCl. Suggested holding time for the extraction fluid is 2 days.
8.5 PREP Extraction Fluid: Add 8.3mL of HCl to 991mL UPW (0.1M HCl). Homogenize. Adjust pH to 2 using Potassium Dihydrogen Phosphate crystals. Suggested holding time is 2 days.

9.0 QA/QC

9.1 An initial demonstration of capability (IDC) study will be performed at the request of specific projects.
9.2 A demonstration of capability (DOC) study will be performed at the request of specific projects.
9.3 A method detection limit (MDL) study is not required for the PBEP.
9.4 A LOD/LOQ verification study will be performed at the request of specific projects.
9.5 Matrix investigation samples, (MS/MSD) are prepared at a rate of 1 set per 20 field samples or one per analytical batch. MS/MSD samples should be chosen randomly from a client batch of samples unless they are pre-selected by the client. Analysts should rotate the client selected for matrix spikes so that recovery and precision data is collected from a wide variety of sample matrices. An MS/MSD sample pair should be processed with each analytical batch if there is sufficient sample. If sufficient sample is not provided then the LCS/LCSD sample results will be used to evaluate analytical batch recovery and precision. This should be noted in the case narrative. Poor recoveries may indicate matrix interference from the sample and should be reported to the client whose sample was used to prepare the MS/MSD. MS/MSD source is the same as the primary calibration standards.
9.6 One method blank and one blank spike are included in each batch of 20 samples. LCS source is the same as the primary calibration standards.
9.7 Duplicates are analyzed at a frequency of 5% when it is not possible to perform a MS/MSD or if the client specifically requests duplicate data. The acceptance criteria for duplicate analysis is <20%RPD.
9.8 Uncertainty of measurements: The uncertainty of measurements shall be calculated by following ASL SOP30.
9.9 The major sources of uncertainty in this method are contamination of vessels, extraction fluid or pipette tips. Contamination problems can be avoided by pre-rinsing vessels and pipette tips three times in 5% HNO3.

10.0 PROCEDURE

10.1 See Table 1 for a guide to the various procedures used by ASL for various clients requesting the PBEP.
10.2 Dry samples according to Table 1.
10.3 Subsampling prior to analysis will be completed following the procedures in the SOP on Subsampling of Soil, Water, and Mixed Matrices ASL SOP40.
10.4 Pre-clean the sieve using 1 + 4 nitric acid and 3 rinses of UPW. Dry the sieve. Sieve samples to <250um generating enough to perform PBEP and total digestion according to SOP 10.
10.5 Turn on the hot bath and equilibrate to 37 $\pm$ 2°C.
10.6 Bring pH meter into lab near the hot water bath and calibrate
10.7 Prepare the appropriate extraction fluid.
10.8 Weigh dry, sieved soil into a small weigh boat. Record the mass on the bench sheet and protect the measured soil sample by placing another weigh boat upside down over the top.
10.9 Measure the extraction fluid.
10.10 Place soil sample into the extraction vessel.
10.11 Pour extraction fluid into the extraction vessel using some of it to rinse the empty weigh boat.
10.12 Add 0.50mL of spike solution to the LCS and MS/MSD.
10.13 Immediately place into the hot water bath and record the start time and temperature.
10.14 If Table 1 indicates that pH adjustment is required, check the pH of the extraction fluid at 5, 10, 15, and 30 minutes. Adjust pH drop wise (usually 5 drops) with HCl to maintain the pH at 1.5 $\pm$ 0.5. Usually pH adjustment is only required at 5 minutes – sometimes 10minutes.
10.15 If Table 1 indicates that agitation is required, manually agitate every 5 minutes or in one case add argon aeration at the rate of 1L/minute.
10.16 Remove samples from the hot water bath after 1 hour. Record the final time, temperature, and pH. Final pH should be taken immediately while temperature is still 37 $\pm$ 2°C.
10.17 If Table 1 indicates filtration is required, filter samples through a pre-cleaned 0.45 um filtration apparatus. If Table 1 indicates that centrifugation is required, centrifuge to separate the soil from the aqueous phase. Decant the aqueous phase.
10.18 Discard the soil phase. Store the aqueous phase in polyethylene vessels. Preserve with nitric acid if required.
10.19 Refer to Table 1 for storage conditions.

11.0 DATA REDUCTION

Transcriptions of hand generated data are reviewed by the analyst when entered into the electronic reporting system.

12.0 DOCUMENTATION

12.1 Preparation of the extraction fluid must be documented in the Metals Standards/Reagent Log Book.
12.2 Extraction Log must be completed and submitted with the extracts to the ICP/ICPMS lab. Locate an extraction log at G:\Controlled Forms\metals.
12.3 After a one year period, all data is archived to the record center for a time totaling 7 years from the date of analysis.

13.0 REFERENCES

13.1 Mark E. Kelley, Susan E. Brauning, Rosalind A. Schoof, and Michael V. Ruby. "Assessing Oral Bioavailability of Metals in Soil"
13.2 Solubility/Bioavailability Research Consortium. "Standard Operating Procedure: In Vitro Method for Determination of Lead and Arsenic Bioaccessibility."
13.3 Ruby, M.V., A. Davis, T.E. Link, R. Schoof, R.L. Chaney, G.B. Freeman, and P. Bergstrom. 1993. "Development of an In Vitro Screening Test to Evaluate the In Vivo Bioaccessibility of Ingested Mine-Waste Lead." Environmental Science and Technology 27(13):2870-2877.
13.4 Ruby, M.V., A. Davis, R. Schoof, S. Eberle, and C.M. Sellstone. 1996 "Estimation of Lead and Arsenic Bioavailability using a Physiologically Based Extraction Test." Environmental Science and Technology 30(2):422-430.

14.0 DEFINITIONS

14.1 ASL – Applied Sciences Laboratory
14.2 CVO – Corvallis, OR
14.3 SBRC-Solubility/Bioavailability Research Consortium
14.4 PBET-Physiologically Based Extraction Test
14.5 PREP-Physiologically Relevant Extraction Procedure
14.6 Bioaccessibility-the fraction of a chemical solubilized from a soil sample using in vitro test methods that simulate gastrointestinal conditions. As per the SBRC, the in vitro method has correlated will with relative bioavailability values – for lead-bearing soils tested in the U.S. EPA swine studies.
14.7 Bioavailability-the extent to which a chemical can be absorbed by a living organism
14.8 Relative bioavailability-a measure of the difference in extent of absorption among two or more forms of the same chemical (e.g. lead carbonate vs. lead acetate), different vehicles (e.g., food, soil, water), or different doses.
14.9 Absolute bioavailability-the fraction of a chemical which is ingested, inhaled, or applied on the skin surface that actually is absorbed and reaches the systemic circulation.
14.10 NELAC – National Environmental Laboratory Accreditation Conference
14.11 NELAP – National Environmental Laboratory Accreditation Program
14.12 QA/QC – Quality Assurance/Quality Control
14.13 QA – Quality Assurance
14.14 QC – Quality Control
14.15 SOP – Standard Operating Procedure
14.16 IDC – Initial Demonstration of Capability
14.17 RSD – Relative Standard Deviation
14.18 %D – Percent Difference
14.19 LCS – Laboratory Control Standard
14.20 LCSD – Laboratory Control Standard Duplicate
14.21 Internal Standard (IS) – A pure analyte(s) added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other method analytes and surrogates that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component.
14.22 Laboratory Duplicates (Dup) – Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analysis of duplicates indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.
14.23 Field Duplicates (FD) – Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedure. Analyses of Duplicates gives a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.
14.24 Laboratory Replicates – An aliquot of sample is taken in the laboratory and prepared. The prepared sample is then analyzed twice. Laboratory replicates indicate precision associated with instrumentation and not sample preparation. For some test methods, a laboratory duplicate and a laboratory replicate may be the same thing.
14.25 Laboratory Reagent Blank (WB1, SB1, XB1) – An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The blank is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
14.26 Blank Spike (BS1W, BS1S) – An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.
14.27 Matrix Spikes/Spike Duplicates (MS/MSD) – An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS/MSD is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

14.28 Stock Standard Solution (SSS) – A concentrated solution containing one ore more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

14.29 Primary Standard Solution (PSS) – A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.

Table 1

	SBRC protocol (HDOH)*	**Bioaccessibility of Ingested Mine-Waste Lead**	**Physiologically Based Extraction Test (PBET)**	**Physiologically Relevant Extraction Procedure (PREP)**
Drying	Air Dry	Air Dry	50ºC 24 hours	65ºC
Sieve Size	<250um	<250um	<250um	<250um
Extraction Fluid	0.4M glycine, adjusted to pH1.5 with HCl	Pepsin, acetate, citrate, lactate, malate	Pepsin, acetate, citrate, lactate, malate	0.1M HCl, adjusted with 0.1M phosphate buffer
pH of Ext Fluid	1.5	1.3	1.3	2.0
Mass (g)	1 ± 0.05	4 ± 0.01	0.4	1
Volume (ml)	100 ± 0.5	40	40	30
Temperature (°C)	37 ± 2	37 ± 2	37 ± 2	37 ± 2
pH adjustment	yes	yes	yes	no
Extraction	agitate	agitate	aerate with argon	agitate
Filtration	0.45um	no	no	no
Centrifugation	no	yes	yes	yes
Preserve	no	no	no	yes
Storage	4ºC	RT	RT	RT
Holding Time	180 days	180 days	180 days	180 days

*Solubility/Bioavailability Research Consortium protocol is recommended by the Hawaii Department of Health and requested most often by Hickam AFB projects.

CHANGE HISTORY

1. Changes made in revision 2

1.1 Section 7.0: Add reference to the Purchasing SOP and Metrology/Equipment matrix.
1.2 Section 8.0: Add statement regarding the verification of standards.
1.3 Sections 9.8 and 9.9: Separation of "Source of error" and "uncertainty SOP reference".
1.4 Section 11.0: Data integrity addressed.
1.5 Removed example of the extraction sheet.

2. Changes made in revision 3

2.1 Updated Cover page
2.2 Section 3.1: Removed second sentence
2.3 Section 3.2: Remove
2.4 Section 9.1, 9.2, 9.4: Change frequency to "will be performed at the request of specific projects"
2.5 Section 9.3: Remove MDL requirement
2.6 Section 12.2: Locate extraction log at G:\controlled forms\metals

3. Changes made in revision 4

3.1 Section 10.0: Added 10.6, pH meter must be near the hot water bath
3.2 Section 10.14: Add HCl drop wise. Usually need to adjust pH at 5 and 10 minutes.
3.3 Section 10.16: Check final pH immediately

Corvallis ASL Standard Operating Procedure

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF METALS BY ICP-MS

METHOD BASED ON THE FOLLOWING SOURCE METHODS:

Preparatory Methods	**Analytical Methods**
EPA 200.2	EPA 200.8 (1994)
SW3010	SW6020 (1994)
SW3010	SW6020A (2007)

APPROVED:

Judy Dykstra

8/11/15

Section Leader — Date

CH2MHILL
Applied Sciences Laboratory (ASL)

SOP Change Form

☐ Temporary Change	**SOP Name:**	**ICPMS**	**SOP No.:**	**MET13**
☒ Permanent Change	**Analytical Batch/SDG:**	**N/A**	**Date:**	**1/14/16**
	Effective Date:	**02/03/16**	**SOP Current Rev.:**	**12**
			Submitted By:	**EMB**
			Approved By:	**JLG**

SOP Section	Change
8.5	Larger volumes of 1000µg/L primary standard may be prepared. 1:100 dilution of stock standards must be maintained.
8.8	Larger volumes of 1000µg/L secondary standard may be prepared. 1:100 dilution of stock standards must be maintained.
8.17	Smaller volumes of working ICSA solution may be prepared. A 1:100 dilution of the stock ICSA standard must be maintained.
8.18	Smaller volumes of working ICSAB solution may be prepared. A 1:100 dilution of the stock ICSA standard and a 1:50 dilution of the 1000µg/L primary standard must be maintained.
8.19	Prepare LLCKs daily.
9.13	An ICV is run immediately after calibration.
9.15 (LLCK)	Add: See Table 4
10.7.2	If IS is added manually to each autosampler vial, it will also need to be added manually to the P/A factor solutions.
Table 4	Note that the LDR standard (or high CCV std) can be run before or after sample analysis
Table 4: MS/MSD note	Remove requirement to inform LPM of MS/MSD failures.

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SOP Change Form

☐	Temporary Change	**SOP Name:**	**ICPMS**	**SOP No.:**	**MET13**
☒	Permanent Change	**Analytical Batch/SDG:**		**Date:**	**3/15/16**
		Effective Date:	**3/15/16**	**SOP Current Rev.:**	**12**
				Submitted By:	**EMB**
				Approved By:	**JLG**

SOP Section	Change
Table 5, Sensitivity	Add criteria for DRC mode: DRC – H2: m/z 89 > 3000 m/z 59 > 500 m/z 78 < 6 DRC – He: m/z 89 > 1500 m/z 59 > 500

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SOP Change Form

☐	Temporary Change	**SOP Name:**	**ICPMS**	**SOP No.:**	**MET13**
☒	Permanent Change	**Analytical Batch/SDG:**		**Date:**	**8/17/16**
		Effective Date:	**8/17/16**	**SOP Current Rev.:**	**12**
				Submitted By:	**EMB**
				Approved By:	**JLG**

SOP Section	Change
Table 4	CCV corrective action: If a CCV associated with DoD samples fails, immediately (within one hour) analyze two additional consecutive CCVs. No samples may be analyzed between the failed CCV and the two additional CCV. If both additional CCVs pass, the samples may be reported without reanalysis. If either fails, recalibrate and reanalyze all samples since last acceptable CCV. Permission from LPM may be obtained to report with flagged failed CCV.

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SOP Change Form

☐ Temporary Change	**SOP Name:**	**DETERMINATION OF METALS BY ICP-MS**	**SOP No.:**	**MET13**
☒ Permanent Change	**Analytical Batch/SDG:**		**Date:**	**9/23/16**
	Effective Date:	**9/23/2016**	**SOP Current Rev.:**	**12**
			Submitted By:	**RM**
			Approved By:	**JLG**

SOP Section	Change
Table 4 Notes ICV/CCV	Break into two different topics, ICV and CCV. ICV: If the ICV fails to meet acceptance criteria, the ICV standard can be re-prepared and re-analyzed once. If the ICV criteria are still failing, then perform instrument maintenance or remake the calibration standards (if necessary) and re-calibrate. CCV: If the CV fails to meet acceptance criteria, perform corrective action such as verifying instrument operation and proper preparation of CV standard. Reanalyze two additional CVs. Both of these CVs must meet acceptance criteria in order for the samples to be reported without reanalysis. If either of these two CVs fail, the analyst will either: 1) Not report the associated samples. Perform additional corrective action and reanalyze the affected samples, or 2) Report the issue to the project LPM who then must obtain and document approval from the client to proceed and note the failure in the case narrative, or 3) If insufficient sample remains for reanalysis, notify the LPM who then must notify the client and document the failure in the case narrative. For non-DoD projects, a single CV may be reanalyzed. If the CV is acceptable, then analysis may continue. If the result is not acceptable, perform additional corrective action, and reanalyze TWO additional CV samples. Both results must be acceptable. If not, recalibration of the instrument is required. Additional corrective action may include routine maintenance or preparation of reagents/standards.
Table 4	Acceptance criteria for method blank for DoD QSM 5.0 are < ½ LOQ, not ½ RL.

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SOP Change Form

☐ Temporary Change	**SOP Name:**	**ICPMS**	**SOP No.:**	**MET13**
☒ Permanent Change	**Analytical Batch/SDG:**	**031617MS-A**	**Date:**	**3/17/17**
	Effective Date:	**3/16/17**	**SOP Current Rev.:**	**12**
			Submitted By:	**EMB**
			Approved By:	**JLG**

SOP Section	Change
8.11	Update concentration of internal standard stock solution to 100µg/mL Sc, Y, In, Tb, Bi.
8.12	Working Internal Standard (2.5 µg/mL): Add 6.25mL internal standard stock and 12.5mL of concentrated nitric acid to approximately 231g of DI water. Alternatively, the internal standard can be added manually. Prepare 1:20 dilution of Stock Internal Standard and add 100µL/10mL sample/standard/QC.

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF METALS BY ICP-MS

1.0 SCOPE AND APPLICATION

This SOP describes the procedures used by CH2M HILL Applied Sciences Laboratory to analyze aqueous and solid samples for selected metals using the Agilent inductively coupled plasma mass spectrometer (ICP-MS). The serial number for the 7500ce is JP14101031. This SOP follows the guidelines of SW-846 6020, 6020A, EPA 200.8, DoD Quality Systems Manual and EPA/625/R-96/010a. The QC requirements for these methods are summarized in Table 4.

2.0 OVERVIEW OF THE ANALYTICAL PROCESS

For the determination of total metals, samples are digested with nitric acid or nitric acid/hydrochloric acid prior to analysis. For the determination of dissolved metals, samples are filtered through a 0.45 um pre-cleaned membrane filter and acidified with nitric acid to pH<2 prior to analysis. For detailed information on sample preparation see the following SOPS: MET10, MET12 and SOP40. Digestates/filtrates are introduced into a radio-frequency argon plasma. Following decomposition, desolvation, atomization, and ionization the ions are extracted through a vacuum interface, separated according to their mass-to-charge ratio by a quadrupole mass spectrometer, and measured by a detector. The following method modifications have been made:

2.1 An instrument detection limit (IDL) study is performed initially and whenever a major instrument change occurs (i.e. new detector).

2.2 EPA 200.8 states the method blank must be <10% of the analyte level found in the samples or <2.2 times the MDL. This SOP states in Table 4 that the method blank must be <RL.

3.0 TARGET ANALYTES, REPORTING LIMITS, AND DETECTION LIMITS

3.1 Refer to Table 1 for a list of target analytes. Current reporting limits can be found in the ASL limits database. Reporting limits are, in general, at least 2 times the laboratory method detection limit.

3.2 Method detection limits (MDLs) may fluctuate from year-to-year based on specific project requirements or instrument performance. The latest MDLs are available from the ASL MDL database upon request. For details on the procedures and criteria used to establish MDLs, consult the *ASL Quality Assurance Program Manual*.

3.3 All reporting limits, QC frequency and QC acceptance criteria are subject to change on a client specific basis as requested by the client.

4.0 INTERFERENCES

4.1 Isobaric elemental interferences – Isobaric elemental interferences occur when an isotope of one element is at the same nominal mass as an isotope of another element (e.g. Mo98 and Ru 98 or Sn114 and Cd 114). Low resolution quadrupole mass spectrometers such as the Agilent 7500ce used in this procedure have insufficient resolution to distinguish between these elements. Of the

elements listed in Table 1, the only masses selected with isobaric elemental interferences are selenium-82 (krypton) and molybdenum-98 (ruthenium). The krypton interference is minimized by use of a high purity source of argon (99.999%) and an interference equation (m/z 82 – 2.76 *m/z 83). The interference on Mo 98 is corrected using the equation in Table 6. The interference on Cd 114 is resolved by reporting a different isotope (Cd111). Correction for isobaric interferences may be made by measuring the intensity due to the interfering element at another isotope and using its natural abundance ratios to correct for its presence at the mass of interest. Common corrections for isobaric interferences are in the Agilent software default method in the section called "interference equations".

4.2 Isobaric polyatomic interferences - Isobaric polyatomic interferences occur when a polyatomic ion formed in the plasma has the same nominal isotope as one of the target elements (e.g. ArCl, m/z=75 interference with As). Polyatomic interferences are controlled by adjusting plasma conditions during tuning to minimize formation of polyatomic ions such as oxides (CeO/Ce is lower when plasma is more "robust") or by using a correction equation (e.g. for cases where Cl is present). Correction equations used are listed in Table 6. The Octopole Reaction System (ORS) employs reactions (hydrogen) and collisions (helium) with gas molecules in the collision/reaction cell to eliminate polyatomic interferences.

4.3 Doubly charged ions – Mid to heavy atomic weight elements (especially the alkaline and rare earths) are prone to forming doubly charged ions that can interfere with the isotopes of interest. Doubly charged ions are created by the loss of two electrons instead of just one. If a doubly charged ion is formed, it will cause a response at half of its elemental mass. The ICP-MS is tuned (using Cerium) to minimize the occurrence of doubly charged ions. For FGD samples, be aware of the following possible interferences: Nd150 or Dy150 on As75 and Gd156 on Se78. Monitor the response of masses 150 and 156. If the cps for these masses is >5 times the cps for the quantitation limit for As or Se, an interference may be possible. Analyze 100ppb Nd, Dy or Gd and measure the response at 150 and 75 or 156 and 78. The ratio can be used to create an interference correction equation (see reference 13.4, section 4.3.3).

4.4 Abundance sensitivity – Abundance sensitivity describes the ability of the mass spectrometer to distinguish between adjacent isotopes that are present in widely different amounts. The Agilent 7500ce has a resolution of about 0.8-amu for peaks that are the same magnitude; however, when a small peak is measured next to an adjacent large peak there may be positive contributions to the small peak from overlap due to the large peak.

4.5 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension may cause physical interferences. Samples with high dissolved solids can cause salt buildup at the tip of the nebulizer (affecting aerosol flow rate) or clogging of the sampler cone. Dissolved solids should be less than 0.2% to reduce possible interferences and sampler cone clogging. Unknown matrices will be tested for conductivity or screened by ICP and diluted appropriately. Internal standards are used to compensate for physical interferences.

4.6 Memory occurs when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from deposits in the sample introduction tubing and from the buildup of sample material in the spray chamber, plasma torch and cones. Memory effects can be minimized by flushing with rinse blanks between samples.

4.7 Contamination can come from pipet tips and autosampler tubes. To prevent contamination, the tips and tubes can be pre-rinsed with 5% HNO_3.

4.8 High TDS samples – The ICPMS is equipped with a High Matrix Introduction (HMI) kit which can be used as an alternative to diluting samples. The HMI modifies the sample introduction system so the sample is diluted with argon prior to the torch. This capability reduces the sample load on the plasma and buildup on the cones. The HMI has several settings (Robust, Ultra Robust (low, medium, high). The MDLs increase with higher argon dilution. See the HMI handbook for instructions.

5.0 SAFETY, WASTE MINIMIZATION, AND POLLUTION PREVENTION

5.1 All samples are assumed to be hazardous and should be handled as such.

5.2 Lab analysts must wear lab coats, safety glasses, and gloves when preparing and handling standards and field and lab samples.

5.3 The persistent presence of noxious odors may be indicative of a failure of the laboratory ventilation system and must be reported to the section lead or laboratory director.

5.4 Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents/standards used in the laboratory.

5.5 Laboratory wastes shall be separated and properly disposed complying with federal, state, and local regulations. The wastes include acid waste, expired sample digestates and stock standards. These wastes shall be handled according to SOP HAZ01, Waste Disposal.

5.6 Analysts are encouraged to reduce the amount of acid waste whenever possible. Neutralize acid waste following SOP HAZ01.

5.7 Safety equipment including a fire extinguisher, first aid kit, eye wash, and chemical spill cleanup kit shall be available for use at all times.

5.8 Analysts are encouraged to reduce the amount of solvent or disposable labware waste whenever possible. More information on this topic can be found in "Less is Better: Laboratory Chemical Management Waste Reduction" from the American Chemical Society.

5.9 Stock standards are purchased in smallest volumes possible to minimize toxic chemicals in house.

6.0 SAMPLE COLLECTION, STORAGE, HOLDING TIMES, AND PRESERVATION

Aqueous samples are collected in clean polyethylene bottles containing HNO_3 as preservative. Samples are transported on ice. The pH of preserved water samples is tested at the time of receipt according to SOP SR02. If the pH of a sample is >2, the sample custodian will notify the client and adjust to pH <2 with nitric acid after client approval. The sample is homogenized and held >24 hours prior to proceeding with digestion or analysis. Samples are stored above freezing to 6°C or at room temperature. Digestates are stored at room temperature. Holding time for metal analyses is 6 months.

7.0 APPARATUS AND MATERIALS

All purchasing of apparatus, materials, standards, gases, and reagents is completed according to the SOP31. Support equipment and instrumentation utilized in this SOP and requiring periodic metrological verifications are tracked in ASL's Metrology/Equipment database.

- Inductively Coupled Plasma Mass Spectrometer - Agilent 7500ce
- Dedicated PC with Windows XP professional and ICP-MS ChemStation software
- Autosampler Tubes and caps
- 50mL digestion vessels
- ISTD tubing; ID 0.19mm (red Tygon®) or ID 0.25mm (orange/blue Tygon®)
- Sample tubing; ID 1.02mm (white/black or white/white Tygon®)
- Drain tubing; ID 1.52mm (yellow/blue Santoprene®)
- Quartz Torch, 2.5 mm injector
- Spray Chamber, Quartz, 90°
- Nickel Sampler Cone
- Nickel Skimmer Cone
- Micromist Nebulizer
- Babington Nebulizer
- Analytical balance readable to 0.01-g

8.0 STANDARDS, GASES, AND REAGENTS

Stock standards are assigned a unique number when received. A copy of the Certificate of Analysis is placed in the ASL chemical inventory database. Primary and Secondary standards are assigned a unique

number when prepared, expire on the expiration date of the stock standard or 6 months from the preparation date, whichever is sooner. Store at room temperature. Standards prepared in the laboratory contain 5% HNO_3. Calibration standards are prepared daily. Verification of the primary and secondary standards is recorded in the standard logbook.

8.1 Liquid argon, hydrogen and helium (99.999%)
8.2 Concentrated nitric acid, trace metals grade
8.3 Ultrapure water (UPW)
8.4 Stock Standards – Purchased. See Table 3. ICV stock is from a different vendor than the stock used for the primary standards.
8.5 Primary Standard (1000 µg/L) – See Table 3
Pipette 9.8mL dilution water (5% HNO3) into an autosampler tube. Add 0.100-mL Stock 1 and 0.100-mL Stock 2 using a pipette. Cap and mix well.
8.6 Calibration Standards - See Table 3.
8.6.1 *CAL STD* - Pipette 29.4 mL dilution water (5% HNO3) into a 50mL digestion vessel. Add 0.600 mL Primary Standard using a pipette. Cap and mix well. For Lithium (10.00-µg/L) - Pipette 9.9mL dilution water (5% HNO3) into an autosampler tube. Add 0.100-mL of Primary Standard using a pipette. Cap and mix well.
8.6.2 *CAL BLK* – Add 237.5mL of UPW (straight from system) into the Blank Bottle. Dispense 12.5mL HNO_3, cap and mix well. Pour into 50mL digestion vessel.
8.7 ICB/CCB
Use the blank prepared in 8.6.2.
8.8 Secondary Standard (1000 µg/L) – See Table 3
Pipette 9.8 mL dilution water (5% HNO3) into an autosampler tube. Add 0.100-mL of both ICV Stock Standards using a pipette. Cap and mix well.
8.9 Initial Calibration Verification (ICV) Standard. See Table 3
Pipette 9.9 mL dilution water (5% HNO3) into an autosampler tube. Add 0.100-mL of Secondary Standard using a pipette. Cap and mix well. For Lithium (1.00-µg/L), Pipette 99.9mL of dilution water (5% HNO3) into a container. Add 0.100-mL Secondary Standard using a pipette. Cap and mix well.
8.10 Continuing Calibration Verification (CCV) Standard. See Table 3.
Same solution as 8.6.1.
8.11 Stock Internal Standard - (Bi, In, Sc, Tb, and Y at 20-mg/L), 2008ISS.
8.12 Working Internal Standard (1-mg/L) – Dilute 2.0-mL Stock Internal Standard and 10.0-mL HNO_3 to 200-mL with UPW. Alternatively, internal standard can be added manually. Prepare 1:20 dilution of Stock Internal Standard and add 100uL/10mL sample/standard/QC.
8.13 Tune Standard Stocks
8.13.1 SW6020 Tune solution, (10-µg/mL Li, Tl, Co, In)
8.13.2 Single Element Ce and Y (10,000µg/ml)
8.13.3 EPA 200.8 Tune solution (10-µg/L Be, Co, In, Pb, Mg)
8.14 Working Tune Solution for Agilent Tune - (1-µg/L Li, Y, Ce, Tl, Co, In) – Dilute 0.10-mL of Ce and Y to 10-mL with dilution water (5% HNO3). Tare empty container and add ~100mL UPW. Add 0.50-mL SW6020 Tuning solution (10-µg/mL Li, Tl, Co, In), 0.50-mL 10-µg/mL Ce and Y and 25.0-mL HNO_3. Bring to 500g with UPW. Dilute solution to1-µg/L using 5% HNO3.
8.15 Working Tune Solution for EPA 200.8 and SW6020 (50ppb Be, Pb, Mg, Li, Tl and 100ppb Co, In). Tare empty container and add ~20mL UPW. Add 1.0-mL SW6020TS, 1.0-mL 2008TS and 10.0-mL HNO_3. Bring to 200g with UPW.
8.16 Stock ICSA/ICSAB Standard - Purchased from Inorganic Ventures
ICSA (6020ICS-0A): 10,000µg/mL Cl, 2000 µg/mL C, 1000 µg/mL Al, Ca, Fe, Mg, P, K, Na, S and 20 µg/mL Mo, Ti in 3.5% HNO_3.
8.17 Working ICSA solution – Tare empty container and add ~20mL UPW. Add 0.5-mL ICSA Stock and 2.5-mL HNO_3. Bring to 50g with UPW. Maintain dissolved solids less than 0.2% to reduce sampler cone clogging. Prepare fresh weekly.

8.18 Working ICSAB solution – Tare empty container and add ~20mL UPW. Add 0.5-mL ICSA Stock, 1.0-mL 1000ppb Primary Standard, and 2.5-mL HNO_3. Bring to 50g with UPW. Maintain dissolved solids less than 0.2% to reduce sampler cone clogging. Prepare fresh weekly.

8.19 LLCK (Low Level Check) – A standard at or below the reporting limit. Dilute Calibration Standard as needed and define preparation on the dilution page included with the raw data.

9.0 QA/QC

9.1 Instrument Tuning: The ICP-MS must be tuned prior to calibration. The instrument tune will be checked daily against the criteria in Table 5 and the instrument will be retuned using auto or manual tune as needed. The instrument will be re-tuned after major maintenance (e.g. cleaning the ICP-MS) or changes in the configuration. (e.g. different spray chamber, shield torch use).

9.2 Initial Demonstration of Capability (IDC): An IDC must be performed whenever there is a significant change to instrument type or personnel. Four solutions of the same formulation are analyzed. The acceptance criteria for the IDC is the acceptance criteria of the blank spike (Table 4).

9.3 Demonstration of Capability (DOC): A DOC must be performed once per year per analyst. Successful analysis of the Performance Evaluation Sample can serve as a DOC.

9.4 Method Detection Limit (MDL): A MDL study should be performed by analyzing 7 replicates of a low level standard. It will be necessary to prepare various solutions to complete the MDL for all the elements. See the ASL QAPP for additional information. MDL studies are performed initially and whenever a major instrument change occurs. MDL studies are performed annually on a project specific basis as requested by the QAPP.

9.5 Linear Dynamic Range (LDR): Twice yearly (DoD) a LDR study must be performed by analyzing solutions of increasing concentrations. The LDR is the highest level analyzed that is within 10% of the true value. Results of the latest LDR study can be found at G: METALS\LDR. If the sample concentration is greater than 90% of the LDR, it is diluted and re-analyzed. For DoD work, if a sample result exceeds the concentration of the calibration standard, analyze a LDR solution (also called a high level CCV) that exceeds the sample result.

9.6 Method Blank/Laboratory Control Spike (LCS): One method blank and one blank spike are included in each analytical batch of 20 samples. LCS source is the same as the calibration standards. The LCS acceptance criteria are listed in Table 4.

9.7 Matrix Spike/Matrix Spike Duplicate (MS/MSD): Spikes are rotated among sample types and clients and analyzed at a frequency of 5%; however, if there are >10 samples in a digestion batch that require 200.8 analyses (excluding treatability), include both ICP and ICPMS MS/MSD spikes in the analytical batch, remembering to dilute the ICP spikes. If it is not possible to analyze the ICP MS/MSD, then include a note in case narrative (i.e., MS/MSD performed on 5% rather than 10% of client samples). Samples are spiked using a primary source and the spike includes all reported analytes.

9.8 Duplicate (D): Duplicates are analyzed at a frequency of 5% when it is not possible to perform a MS/MSD or if the client specifically requests duplicate data. The acceptance criteria are listed in Table 4.

9.9 Dilution Test/Post Spike (DL/PS): Serial dilution test samples (1:5 dilution) and post digestion spikes are analyzed once per batch when performing SW6020 and DoD methods.

9.10 Uncertainty of measurements: The uncertainty of measurements shall be calculated by following ASL SOP30.

9.11 The major sources of uncertainty in this method are preparation of calibration/calibration verification solutions, contamination of tubes or pipette tips, and sample introduction problems. Contamination problems can be avoided by soaking autosampler tubes in 5% HNO3 and pre-rinsing pipette tips three times in 5% HNO3 prior to use.

9.12 Limit of Detection/Limit of Quantitation (LOD/LOQ): See SOP 32. LOD and LOQ verifications are performed quarterly for DoD,

9.13 Initial Calibration Verification (ICV): An ICV must be analyzed prior to sample analyses using a second source standard.

9.14 Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB): A CCV/CCB must be analyzed between every ten analyses and after the last sample.

9.15 Low Level Check Standard (LLCK): A LLCK must be analyzed after calibration. As per page 214 of the 2003 NELAC Standard, "A standard corresponding to the limit of quantitation must be analyzed with each analytical batch and must meet established acceptance criteria." Low level check standards are analyzed with each batch and must be 50-150% of the true value if no requirement is listed in a project-specific QAPP.

9.16 Interference Check Standard A and B (ICSA/ICSAB): ICSA/ICSAB are analyzed at the beginning of an analytical run when following SW6020 and DoD QSM.

9.17 Internal Standard (IS): The expected values of the internal standards are the original responses of the internal standards in the daily calibration blank.

9.18 Direct Analysis: Drinking water samples (turbidity <1NTU) and Non-DoD filtered/preserved samples requiring dissolved analyses are analyzed directly after preparing as follows: Add 10mL of sample and 0.5mL of HNO3 to the autosampler tube. Homogenize and analyze using 1.05 as the dilution factor. Prepare associated QC as follows: WB=10mL UPW + 0.5mL HNO3, BS=9.8mL UPW, 0.20mL working stock standard (8.5) and 0.5mL HNO3, MS/MSD=9.8mL sample, 0.20mL working stock standard (8.5), and 0.5mL HNO3. Homogenize.

10.0 PROCEDURE

10.1 Cold Start-Up (after being unplugged for a move, maintenance, or long-term shutdown)
- 10.1.1 Ensure the instrument and all accessories (pumps, hoses, etc.) are all connected.
- 10.1.2 Turn on the rotary pump power switch. Once the switches are manually on they can be controlled by the ChemStation software.
- 10.1.3 Turn on the main power breaker and the pump switch on the rear of the instrument.
- 10.1.4 Turn on the instrument power switch on the front panel.
- 10.1.5 Turn on the printer and computer and start the ChemStation software.
- 10.1.6 Select *Instrument>>Instrument Control*.
- 10.1.7 Continue with 10.2 Starting from Shutdown Mode.

10.2 Starting from Shutdown Mode (Vacuum off, e.g. after MS maintenance)
- 10.2.1 Select *Vacuum>>Vacuum On.* Click *Yes* when the dialog box appears asking if you want the vacuum on. The instrument will turn on the rotary pump, open the backing line valve and then turn on the turbomolecular pump. The Instrument Control display will indicate that the rotary pump is on. Depending on the length of the shutdown it will take anywhere from 15 minutes to 2 hours for the vacuum chamber to reach the correct pressure of 5×10^{-4} Pa.
- 10.2.2 Once the pressure reaches 5×10^{-4} Pa the instrument is in Standby Mode.

10.3 Starting from Standby Mode (normal mode between sample batches)
- 10.3.1 Make sure that the title bar shows *Standby* Mode.
- 10.3.2 Turn on the water recirculator. The Ar pressure should be 680-720 kPa. Cooling water flow should be 1.3-5 L/min.
- 10.3.3 Check the drain carboy and all tubing connections. Make sure the peristaltic pump tubing is in good condition and are clamped down. Change the pump tubing if needed. Move the sample probe to the rinse bottle and place the internal standard line in a 5% nitric acid solution.
- 10.3.4 Select Plasma>>Plasma On. Click Yes when the dialog box appears asking if you want the plasma on. The gases are switched on, and lines purged before the plasma ignites. After the plasma ignites the interface chamber will be evacuated.
- 10.3.5 The instrument title bar will show *Analysis* mode.
- 10.3.6 Double check that the drain is flowing by observing the flow in the tubing.

10.4 Check the Instrument Operation
- 10.4.1 Select *Instrument>>Instrument Control*. The instrument control window will appear with a schematic of the instrument. Placing the cursor over a gauge (e.g. Ar Tank Pressure) and then left clicking will show the reading for that gauge.
- 10.4.2 Monitor the analyzer pressure, water flow, water temperature.

10.5 Instrument Tune as per Agilent

10.5.1 After instrument warmup of at least 10 minutes, aspirate the tuning solution 1-ppb Li, Y, Ce, Co, In and Tl. Check tune results in the tune window against the criteria in Table 5, and adjust parameters as needed. Tune the instrument after maintenance (cone cleaning) or changes in the configuration (different spray chamber or torch). Auto tune of the detector can be performed two times/wk.

10.6 Instrument Tune as per EPA 200.8 and SW6020.

Working Tune Solution for EPA 200.8 and SW6020 is analyzed prior to calibration. Acceptance criteria and parameters to adjust are found in Table 5.

10.7 P/A Factor Adjustment

10.7.1 The P/A factor is used to ensure linear calibration curves are obtained when switching between the pulse and analog mode calibration curves. To gather accurate P/A factors the element counts must be in the 400,000 to 4,000,000 cps range. This is accomplished by using standards containing each target element (suggested range 10-100ppb)

10.7.2 After completing the tune check in section 10.5, the internal standard line is moved from the 5% nitric acid solution to the internal standard solution. Before setting the P/A factors, make sure the internal standard solution has reached the nebulizer. Select *Tune>>P/A Factor*. Place the P/A solution in the default vial position found in the window. Select *Run*. When completed (several minutes) accept the P/A factors which were found and repeat the process with various concentrations in order to find P/A factors for the majority (>80%) of the analytes. After analysis of the first P/A solution be sure to check the box for "Merge in current data."

10.8 Selecting a Method

10.8.1 Select *Method>>Load.* Select the appropriate method from the list. Select "NOGAS.M" to run the instrument without using the reaction cell (DRC). DRC methods include Hydro.M (selenium) and Helium.M (other CC elements in Table 2). HMI methods include He-HMI.M, HydroHMI.M, and HeHy-HMI.M.

10.8.2 A window will allow you to select a calibration file. Select OK to default calibration file.

10.9 Setting up an Analytical Sequence

An analytical sequence is used to automatically analyze a series of standards and/or samples. Select *Sequence>>Load*. Select the template. Select *Sequence>>Edit Sample Log Table.* The sample log table for the sequence currently in memory will be opened. Modify this sequence as needed and save it under a new name. See Table 4 for guidance in setting up a proper sequence.

10.10 Preparing samples for analysis

It is recommended that for each new or unusual matrix, a semi-quantitative analysis is performed using the ICP to screen for elements at high concentration. This may be useful for preventing damage to the instrument and predicting required dilutions. In general, it is recommended that the concentration of Ca, Mg, Fe, Al, Na, and K be kept below 200,000ppb. The ICPMS can also be conditioned by injecting a high matrix sample at a dilution for about 10 minutes prior to calibration.

10.11 Running a Sample Analysis

10.11.1 Load the samples and/or standards into the appropriate positions on the autosampler.

10.11.2 Select *Sequence>>Run*. From the Start Sequence dialog box select Full Method and Overwrite Existing Data Files. Enter your initials under Operator Name. Use the Default Data file directory that is automatically created. This system creates names based on the current time and date. The format is year, month (A-L are 1-12), day, hour (A-X is 0-23), and an alphanumeric (00, 01, 02 … zy, zz).

10.11.3 Click Run Sequence.

10.11.4 The curve is generated after the tune samples. The calibration solutions are analyzed as directed in the sequence. Select *Sequence>>Edit Sample Log Table.* Under Sequence Flow click on the "Calib" block. The two calibration standards are listed here (Type=CalStd) and distinguished as Level 1 & 2 (Data File=cal0 and cal4).

10.11.5 The calibration curve fit Y=aX + b and units, µg/L are to be selected in the method. (If using a 2 point curve with one being the calibration blank, Y=aX + b and Y=aX + [blank] give the same result.)

10.11.6 The internal standards in the method are to be set as shown in Table 2.

10.11.7 Dilute the samples if the concentration is >90% of the current Linear Dynamic Range.
10.11.8 For DoD work, analyze a LDR solution (also called a high level CCV) that exceeds the sample result.

11.0 DATA REDUCTION

11.1 Calculation of cations concentration in aqueous samples

Conc. (µg/L) = Conc. of digestate (µg/L) x dilution factor

11.2 Calculation of matrix spike recoveries

$$\%R = \frac{\textit{Spiked sample result - Native sample results}}{\textit{Spike amount added}} \textit{ x 100}$$

11.3 Calculation of LCS recoveries

$$\%R = \frac{\textit{Blank spike result}}{\textit{Spike amount added}} \textit{ x 100}$$

11.4 Calculation of relative percent difference

RPD = (Difference/Average) x 100

11.5 Calculation of internal standard recoveries (by ChemStation software)

$$\%R = \frac{\text{counts per second ISTD in sample}}{\text{counts per second ISTD in calblk}} \text{x}100$$

11.6 Calculation of sample concentration (by ChemStation software). The mean counts per second (CPS) from the initial calibration analysis is used to calculate the concentration in the sample. The equation for determining concentration is as follows:

$$\text{Sample concentration} = \frac{\text{A * concentration of standard}}{\text{B}} * \text{DF}$$

Where: A = mean CPS of the sample for the element to be measured
B = mean CPS of the calibration standard for the element to be measured
DF = dilution factor

12.0 DOCUMENTATION

12.1 A hard copy of the analytical results and run log will be generated and filed in the ICP/ICPMS filing cabinet for a period of one year. After a one year period, all data is archived to the record center for a time totaling 7 years from the date of analysis.
12.2 Standards that are prepared and used in one analytical run (i.e. calibration standard, ICV/CCV and LLCK standards) are recorded on the dilution/post spike summary sheet. All other standard preparation is documented in the Metals Working Standard Log Book.

12.3 Maintenance issues are entered into the ICP-MS Maintenance Log Book.
12.4 All data is stored on the computer hard drive after sample analysis. Every 24 hours during the business week (not on weekends), the local hard drives responsible for collecting instrument data are backed up to a RAID 5 server dedicated for this purpose. On a monthly basis, the server is backed up to DVD. This will remain in storage off site for at least 7 years from the date of the original data creation.
12.5 Turbidity results and acid used to matrix match are recorded on a Metals Digestion Benchsheet located at G:\\controlled forms\Metals.

13.0 REFERENCES

13.1 Method for the Determination of Metals in Environmental Samples, U.S.EPA Method 200.8, Revision 5.5 (1994)
13.2 Test Methods for Evaluating Solid Waste, EPA SW-846 Method 6020, 6020A and Chapter 3 -- "Metallic Analytes"
13.3 DoD Quality Systems Manual for Environmental Laboratories Version 5.0, 4/22/2009
13.4 FGD ICP/MS Standard Operating Procedure: Inductively Coupled Plasma/Mass Spectrometry for Trace Element Analysis in Flue Gas desulfurization Wastewaters, U.S.EPA (Draft May 2011)
13.5 HMI Handbook, Agilent 7500 Series ICPMS

14.0 DEFINITIONS

14.1 ASL – Applied Sciences Laboratory
14.2 CVO – Corvallis, OR
14.3 NELAC – National Environmental Laboratory Accreditation Conference
14.4 NELAP – National Environmental Laboratory Accreditation Program
14.5 AFCEE QAPP- Air Force Center for Environmental Excellence Quality Assurance Project Plan
14.6 CLP – Contract Laboratory Program
14.7 QA/QC – Quality Assurance/Quality Control
14.8 QA – Quality Assurance
14.9 QC – Quality Control
14.10 ICP – Inductively Coupled Plasma
14.11 SOP – Standard Operating Procedure
14.12 IDC – Initial Demonstration of Capability
14.13 DOC – Demonstration of Capability
14.14 DRC – Dynamic Reaction Cell
14.15 LDR – Linear Dynamic Range
14.16 RSD – Relative Standard Deviation
14.17 MDL – Method Detection Limit
14.18 MS – Mass Spectrometer
14.19 RL – Reporting Limit
14.20 CRDL – Contract Required Detection Limit
14.21 CRI – A standard for the ICP that contains elements at the CRDL
14.22 IPC – Instrument Performance Check
14.23 SIC – Spectral Interference Check
14.24 UPW – Ultrapure Water
14.25 %D – Percent Difference
14.26 LCS – Laboratory Control Sample
14.27 Laboratory Duplicates (Dup) – Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of duplicates indicate precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.
14.28 Laboratory Reagent Blank (WB1, SB1, XB1) – An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The blank is used to

determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

14.29 Initial/Continuing Calibration Verification (ICV/CCV) – A solution used to evaluate the performance of the instrument system with respect to a defined set of method criteria. The ICV is obtained from a source different from the source of calibration standards. Same as QCS.

14.30 Initial/Continuing Calibration Blank (ICB/CCB) – A reagent blank consisting of UPW and nitric acid analyzed after every ICV/CCV.

14.31 Blank Spike (BS1W, BS1S) – An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

14.32 Matrix Spikes/Spike Duplicates (MS/MSD) – An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS/MSD are analyzed exactly like a sample, and their purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

14.33 Stock Standard Solution (SSS) – A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

14.34 Primary Standard Solution (PSS) – A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.

14.35 Calibration Standard (CAL) – A solution prepared from the primary standard solution or stock standard solution and the internal standards and surrogate analytes. The Cal solutions are used to calibrate the instrument response with respect to analyte concentration.

Table 1
Target analyte list, analytical isotopes, Interferences, and isotopic abundances

Element	Mass Isotope	Possible Interferences	Stable Isotopes (abundance)
Al	27		27 (100)
Sb	121	$^{105}Pd^{16}O$, $^{9}Y^{16}O_2$	121(57.4), 123(47.6)
As (and CC)	75		75 (100)
Ba	137		137 (11.2)
Be	9		9 (100)
Cd	111		106(1.3), 108(0.89), 110 (12.5), 111 (12.8), 112 (24.1), 113(12.2), 114(28.7), 116(7.5)
Cr (and CC)	52	$^{36}S^{16}O$, $^{36}Ar^{16}O$	50(4.3), 52(83.8), 53(9.5), 54(2.4)
Co (and CC)	59		59 (100)
Cu (and CC)	63		63 (69.2), 65 (30.8)
Pb	208	$^{192}Pt^{16}O$, $^{192}Os^{16}O$	204(1.4), 206(24.1), 207(22.1), 208(52.4)
Li	7		7 (92.5), 6 (7.5)
Mn (and CC)	55		55 (100)
Mo	98		98 (24.1), 96 (16.7)
Ni (and CC)	60		58 (68.1), 60(26.2), 61 (1.1), 62(3.6), 64(0.9)
Se	82	$^{12}C^{35}Cl_2$	74(0.9), 76(9.4), 77(7.6), 78(23.8), 80(49.6), 82(8.7)
Se (CC only)	78	NA	78(23.8),
Ag	107	$^{91}Zr^{16}O$	107(51.8), 109(48.2)
Tl	205	$^{189}Os^{16}O$	203(29.5), 205(70.5)
V (and CC)	51		51 (99.75)
Zn	66		66(27.9), 67(4.1), 68 (18.8), 70(0.6)

**The ^{50}Cr, ^{53}Cr, ^{54}Cr lines suffer from many more potential interferences from sulfur, chlorine, and argon compounds of oxygen, nitrogen, and carbon.

Table 2
Internal Standards

ISTD	Mass	Interferences	Applicable Elements
Scandium	45	$^{16}O_2{}^{12}CH$, $^{29}Si^{16}O$, $^{90}Zr^{+2}$	Li, B, Al, V, Be, Cr
Yttrium	89	$^{73}Ge^{16}O$, $^{178}Hf^{+2}$	As, Se, Mn, Ni, Cu, Zn, Mo, Co
Indium	115	^{115}Sn, $^{99}Ru^{16}O$	Cd, Ag, Sb
Terbium	159	$^{143}Nd^{16}O$, $^{127}I^{16}O_2$	Ba
Bismuth	209	May be in environmental samples, $^{193}Ir^{16}O$	Tl, Pb

Table 3
Calibration Standards

Element	Stock (mg/L)	Cal Blk (µg/L)	Cal Std (µg/L)	ICV (µg/L)	CCV (µg/L)	Spike (µg/L)
Sb	100	0	20	10.0	20.0	50.0
As	100	0	20	10.0	20.0	50.0
Be	100	0	20	10.0	20.0	50.0
Cd	100	0	20	10.0	20.0	50.0
Cr	100	0	20	10.0	20.0	50.0
Co	100	0	20	10.0	20.0	50.0
Cu	100	0	20	10.0	20.0	50.0
Pb	100	0	20	10.0	20.0	50.0
Li	100	0	2.0	1.0	2.0	5.0
Mn	100	0	20	10.0	20.0	50.0
Mo	100	0	20	10.0	20.0	50.0
Ni	100	0	20	10.0	20.0	50.0
Se	100	0	20	10.0	20.0	50.0
Ag	50	0	10	10.0	10.0	25.0
Tl	100	0	20	10.0	20.0	50.0
V	100	0	20	10.0	20.0	50.0
Zn	100	0	20	10.0	20.0	50.0

Table 4
Analytical sequence and QC requirements for ICPMS analysis

ASL QC Codes	EPA 200.8 Code	Description	EPA 200.8 Revision 5.5	SW-846 6020 and 6020A	DoD QSM
IS		Internal Standard Calibration	60-125% QC 60-125% Samples	80-120% QC 30-120% Samples SW6020A: 70-120%	80-120% QC 30-120% Samples
STDS		Calibration Standards	>0.995 if >1 std used	>0.995 if >1 std used	>0.995 if >1 std used
ICV-DATE	QCS	Initial Calibration Verification	90-110%	90-110%	90-110%
ICB-DATE		Initial Calibration Blank	<RL	<RL	<LOD
LLCK		RL check	50-150%	50-150%	80-120%
ICSA	SIC	Interference Check Sample, mix A	NA	<RL	<LOD
ICSAB	SIC	Interference Check Sample, mix AB	NA	80-120%	80-120%
WB1-DATE	LRB	Method Blank	<RL	<RL	< ½ RL
BS1WDATE	LFB	Blank Spike	85-115%	80-120%	Project Limits. If no project limits, use QSM Appx C limits
XXXX01		Sample 1 native	Dilute if >0.2% solid	Dilute if >0.2% solid	Dilute if >0.2% solid
XXXX01D		Sample 1 duplicate	<20%RPD if >100xMDL	<20%RPD if >100xMDL	<20%RPD
XXXX01MS	LFM	Sample 1 matrix spike	70-130% if spike>30% of sample	80-120% if spike >30% of sample SW6020A: 75-125%	Project Limits. If no project limits, use QSM Appx C limits
XXXX01SD	LFM	Sample 1 matrix spike dup	70-130% if spike>30% of sample and ±20 RPD	80-120% if spk>30% of sample and ±20% RPD SW6020A: 75-125%	±20 RPD
XXXX01DL		Serial Dilution Test	No definition	1:5 Dil ± 10% if smpl >100X rgt blank SW6020A: ± 10% if smpl >10X LOQ after dilution	1:5 Dil ± 10% if smpl >50X LOQ
XXXX01PS		Sample 1 post-spike	No definition	75-125% SW6020A: 80-120%	80-120% if smpl <50X LOQ
CV1-DATE		CCV	90-110%	90-110%	90-110%
CB1-DATE		Continuing Calibration Blank	< RL	<RL	<LOD
LDR		LDR – High CCV (See Below)	NA	NA	90-110%

Method Blank: If the method blank exceeds acceptance criteria, re-pour and re-analyze the method blank. Continue the analytical run if the method blank is <1/10 of any sample or 1/10 the regulatory limit (whichever is greater). Report the data using appropriate qualifiers. Corrective action: Re-prep and re-analyze the batch. If the analyte is a common laboratory contaminant (e.g. aluminum), the acceptance criteria is <RL.

BS: If the BS fails the acceptance criteria, correct the problem (re-prep the analytical batch if necessary) and re-analyze.

ICV/CCV: If the ICV/CCV is outside acceptance criteria, re-analyze. If the CCV is still outside acceptance criteria, correct the problem, recalibrate and re-analyze everything since the last successful CCV.

ICB/CCB: If the CCB is >RL, re-prep/reanalyze calibration blank. For 200.7: if CCB continues to be >RL, terminate analytical run and re-analyze. For SW6020: if CCB continues to be >RL, continue analytical run if sample results are >10x the contamination level. For DoD: if ICB/CCB is >LOD, continue analysis if ICB/CCB is <1/2 RL. Flag ICB/CCB data and note in case narrative. If ICB/CCB is >1/2 RL, re-analyze.

ICSA: For DoD work, all non-spiked analytes <LOD unless verified (i.e. Certificate of Analysis) trace impurity from one of the spiked analytes. To calculate the trace impurities in the ICSA solution, it will be necessary to take into account the dilution used to prepare the solution from the purchased stock solution. If non-spiked analytes >LOD, continue analysis if <1/2 RL. Note in case narrative. If non-spiked analytes >1/2 RL, re-analyze ICSA.

LDR: For DoD work, the LOQ and calibration standard establish the quantitation range (QR). When sample results exceed the QR, analyze a LDR solution (also called a high level CCV) that exceeds the sample result.
Internal Standards: Follow the procedure below if the internal standard out of acceptance criteria is associated with an analyte of interest (Table 2). Analyze the CCB at least once. If the internal standard is within acceptance criteria for the CCB, the internal standard most likely failed due to a matrix interference. Dilute the sample 1:5. If internal standard is outside of acceptance criteria for the CCB, insert one or more wash/rinse samples and make additional sample dilutions until the internal standard is within acceptance criteria. If the internal standard is outside acceptance criteria when analyzing the CCB, correct the problem (i.e. retune the instrument or shut down the instrument to clean cones), re-calibrate and re-analyze all samples after the last successful CCV/CCB.
MS/MSD: If the MS/MSD fails to meet acceptance criteria contact the LPM. If the associated BS/BSD is in control, matrix effect may have caused the spike failure. Note the MS/MSD failure in the case narrative and apply the appropriate flags.

Table 5
Tuning Criteria

Parameter	Criteria	Parameters to Adjust
Stability	200.8: %RSD for masses 9,24,59,115,208 must be < 5% (minimum of 5 integrations) 6020: %RSD for masses 7,59,115,205 must be < 5% (minimum of 4 integrations)	Retune or adjust as needed to meet criteria Check peristaltic pump tubing, make sure instrument has reached thermal stability, check connections, monitor for air bubbles in sample introduction.
Mass calibration	200.8: ± 0.10 amu for masses 24, 25, 26, 206, 207, 208 6020: ± 0.10 amu for masses 7, 59, 115, 205	Retune or adjust as needed to meet criteria Axis Gain – Higher value shifts position of heavier mass toward higher mass. Axis Offset – Higher value shifts position of all masses toward higher mass.
Mass resolution	200.8: <0.8amu @5% peak height 6020: <0.9amu @10% peak height Agilent: 0.7±0.1amu @10% peak height	Retune or adjust as needed to meet criteria AMU Gain – Higher values narrow peak width for heavier masses. AMU Offset – Higher values narrow peak width for all masses.
Oxides	Ratio 156/140 < 2.0%	Retune or adjust as needed to meet criteria Higher RF power increases sensitivity of higher masses and decreases doubly charged ions and oxides Sample depth – longer sample depth decreases oxides and sensitivity Carrier Gas – Lower flows decrease oxide/doubly charged ion levels and decrease sensitivity.
Doubly Charged Ions	Ratio 70/140 < 3.0%	Same as Oxides
Sensitivity	Mass 7 > 3,000 counts Mass 89 > 12,000 counts Mass 205 > 8,000 counts	Retune or adjust as needed to meet criteria Higher RF power increases sensitivity of higher masses and decreases doubly charged ions and oxides. Adjust torch H and V position to adjust sensitivity for all masses. Sample depth – shorter sample depth increases sensitivity and increases oxides Carrier Gas – Higher flows increase sensitivity and increase oxide/doubly charged ion levels. Peri Pump – Higher speed increases sensitivity, too high decreases it.

Parameter	Criteria	Parameters to Adjust
		Higher speeds increase oxide/doubly charged ion levels.

Table 6
Interference equations programmed in ICP-MS software

Element	Correction Equation	Comments
Li(6)	C(6) - C(7)*0.082	
Ca(44)	C(44) - C(88)*0.015	
V(51)	C(51) – 3(C(53) – 0.113*C(52))	Correction for isobaric molecular interference of $^{35}Cl^{16}O^+$ using isotope $^{37}Cl^{16}O^+$. C(52) corrects for ^{53}Cr using ^{52}Cr
Cu(63)	C(63) – C(23)*0.0000555	
Zn(66)	C(66) – C(69)*0.00141	
Zn(67)	C(67) – C(69)*0.034	
Zn(68)	C(68) – C(69)*0.1095	
As(75)	C(75) – 3.127(C(77) – 0.875(C(82) – 1.009*C(83)))	Correction for isobaric molecular interference of $^{40}Ar^{35}Cl^+$ using $^{40}Ar^{37}Cl^+$. C(82) corrects for ^{77}Se using ^{82}Se. C(83) corrects for ^{82}Kr using ^{83}Kr. Note 1: net signal at m/z 82 must be from ^{82}Se only, not BrH^+ Note 2: $^{38}Ar^{37}Cl^+$ contribution is negligible (0.06% of $^{40}Ar^{35}Cl^+$ signal)
Se(77)	C(77) – C(82)*0.874	
Se(82)	C(82) – C(83)*1.009	Correction for isobaric interference of ^{82}Kr using isotope ^{83}Kr
Mo(98)	C(98) – C(99)*0.146	Correction for isobaric interference of ^{98}Ru using isotope ^{99}Ru
Cd(111)	C(111) – 1.073(C(108) + 0.712*C(106))	Correction for $^{95}Mo^{16}O^+$ using $^{92}Mo^{16}O^+$. C(106) corrects for ^{108}Cd using ^{106}Cd. Note 1: C(106) must be from Cd only, not ZrO+ Note 2: An additional isobaric elemental correction should be made if palladium is present.
Cd(114)	C(114) – C(118)*0.027	Correction for isobaric interference of $^{114}Sn^+$ using isotope $^{118}Sn^+$
In(115)	C(115) – C(118)*0.016	Correction for isobaric interference of $^{115}Sn^+$ using isotope $^{118}Sn^+$
Pb (208)	C(208) + C(206) + C(207)	Allows for isotopic variability of Pb

CHANGE HISTORY

1. Changes made in revision 7

1.1 Add review box on cover.
1.2 In Section 8.0, remove recertification statement.
1.3 Section 9.11 include statement of LOQ from NELAC standards
1.4 Update Tables 10.
1.5 EPA 200.8 Revision 5.4 on Table 4 was changed to Revision 5.5.
1.6 Data backup to server is described in section 12.0.
1.7 AFCEE 4.0.01 MDL verification requirement has been added to section 9.4.
1.8 SW6020 modification included in section 2.0
1.9 Section 2.0: MET 02 and MET 15 removed and MET 10 added.
1.10 Removed the procedure to reanalyze the samples if the duplicate or matrix spikes fail in Table 5.

2. Changes made in revision 8

2.1 Section 6: After preserving a sample, hold >24 (instead of 16) hours prior to digestion or analysis.
2.2 Section 9.5: Changed LDR study frequency from 4x/year (AFCEE) to 2x/year (DoD).
2.3 Section 12.0: Record turbidity checks and acid used to matrix match.
2.4 Section 13.0: Add DoD reference.
2.5 Removed Table of PE results. This is not required anymore according to latest NELAC audit.
2.6 Table 4: Acceptance criteria for MS/MSD changed from "<30%" to ">30%".
2.7 Table 4: Changed AFCEE 4.0 column to DoD QSM.
2.8 Table 4: DoD requirement - non-spiked analytes in ICSA must be <LOD. The ICSA note below the table defines this requirement.
2.9 Table 4: DoD requirement - ICB/CCB must be <LOD. The ICB/CCB note below the table provides additional instructions.
2.10 Table 4: Added DoD requirement to analyze LDR solution if sample results are greater than standard as per page 5-16 of DoD QSM.
2.11 Three tables were removed (Acquisition Parameters, Method Conditions and Flowchart for 7500).
2.12 Table for Tuning Criteria was updated.
2.13 Table for Dilutions and Post Spikes updated.

3. Changes made in revision 9

3.1 The approval signature block was updated from the QA officer and the author to the author and the section leader.
3.2 Sections 1.0, 13.2 and Table 4: Add reference to Method 6020A.
3.3 Section 7.0: Add reference to the Purchasing SOP and Metrology/Equipment matrix.
3.4 Section 8.0: Add statement regarding the verification of standards.
3.5 Sections 9.10 and 9.11: Separation of "Source of error" and "uncertainty SOP reference".
3.6 Section 12.5: Add reference to controlled form location.
3.7 Table 6: Removed dilution log form.

4. Changes made in revision 10

4.1 Updated the signature block on cover page
4.2 Section 1.0: Added reference to Table 4
4.3 Section 2.0: Contains reference to digestion SOPs MET10 and MET12
4.4 Keep sections 3.1& 9.1 reference to MDLs. Studies are required for certification in some states
4.5 Section 6: Changed "Samples are stored at 4°C" to "Samples are stored above freezing to 6°C or at room temperature"
4.6 Section 7.0: Updated reference to the Metrology/Equipment database and bullets added

4.7 Sections 7.0 and 8.0: removed vendor names and part numbers
4.8 Section 8.0: Expiration date of standards changed from 6 months to match that of the stock std
4.9 Section 8.0: Differentiated between holding time for Primary/Secondary standards and the calibration standards. E200.8 states "calibration standards should be prepared every two weeks or as needed". Calibration standards at ASL are prepared fresh with each analytical batch
4.10 Section 9.2: Added acceptance criteria for IDC
4.11 Section 10.3.2: change acceptable air flow to 7-8m^3/min
4.12 Section 10.3.3: change acceptable water flow to 1.3-5 L/min
4.13 Table 4: Removed * from ICSA row after "<RL"

5. Changes made in revision 11

5.1 General re-write for entire SOP

6. Changes made in revision 12

6.1 Cover page: Changed SW6020A revision from 1998 to 2007.
6.2 3.2: changed wording regarding MDLs: from "not static and fluctuate yearly" to "may fluctuate annually based on project requirements or instrument performance."
6.3 4.1: added ruthenium interference with molybdenum at mass 98 to isobaric elemental interferences. Referenced equation in table 6.
6.4 4.2: Deleted correction equations. Moved to Table 6. Table 6 created from all equations used in Nogas method currently, even though not all elements listed are reported.
6.5 6.0: Deleted statement that pH of water samples is recorded on sample receipt record.
6.6 8.17, 8.18: added that ICSA and ICSAB should be prepared weekly per SW6020A.
6.7 9.5: changed "quantitation range" to "concentration of cal std"
6.8 10.3.4: deleted that drain carboy should be empty.
6.9 10.7.2: Added that the box for Merging P/A data should be checked.
6.10 10.8.1: added method names.
6.11 10.10: added advice for diluting samples.
6.12 14.14: added DRC to definitions
6.13 Table 4: Added SW6020A criteria (as differs from SW6020) for MS/MSD, PS, and DL.
6.14 Created table 6.

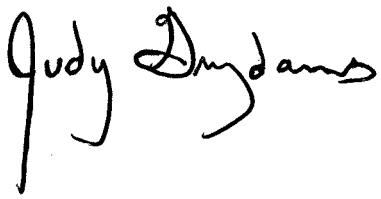
Corvallis ASL Standard Operating Procedure

STANDARD OPERATING PROCEDURE FOR HOT BLOCK DIGESTION OF SEDIMENTS AND SOILS

METHOD BASED ON THE FOLLOWING SOURCE METHODS:

Preparatory Methods	**Analytical Methods**
SW3050B	None

APPROVED:



2/26/15

Section Leader — Date

CH2MHILL
Applied Sciences Laboratory (ASL)

SOP Change Form

☐ Temporary Change	**SOP Name:**	**Digestion of Soils**	**SOP No.:**	**MET10**
☒ Permanent Change	**Analytical Batch/SDG:**	**NA**	**Date:**	**12-8-15**
	Effective Date:	**12-8-15**	**SOP Current Rev.:**	**10**
			Submitted By:	**KMF**
			Approved By:	**JLG**

SOP Section	Change
Section 6.1	Change “Collect soil samples in clean wide mouth glass containers” To, “Collect soil samples in clean plastic or glass containers. (Wide mouth glass containers preferred)”
Section 10.3.20.1	Change “Rinse the graduated cylinder three times with UPW” To, “To clean the graduated cylinder, rinse three times with UPW.”

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SOP Change Form

☐ Temporary Change	**SOP Name:**	**Digestion of sediment and soils**	**SOP No.:**	**MET10**
☒ Permanent Change	**Analytical Batch/SDG:**		**Date:**	**3/25/16**
	Effective Date:	**3/25/16**	**SOP Current Rev.:**	**10**
			Submitted By:	**JLG**
			Approved By:	**JLG**

SOP Section	Change
10.3.8	After "…1:10 dilutions of the 23 element and 7 element QC standards." Add "Alternatively, add 25µL of the 23 element and 7 element QC standards"

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SOP Change Form

☐ Temporary Change	**SOP Name:**	**Digestion of soil samples**	**SOP No.:**	**MET10**
☒ Permanent Change	**Analytical Batch/SDG:**	**NA**	**Date:**	**8-18-16**
	Effective Date:	**8-18-16**	**SOP Current Rev.:**	**10**
			Submitted By:	**KMF**
			Approved By:	**JLG**

SOP Section	Change
3.1	Add the following to the list of target analytes: boron
10.3.20	Change: “Allow digestates to settle overnight prior to analysis.” To: “Allow digestates to settle overnight prior to analysis, or filter as necessary at the instrument for rush samples.”

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SOP Change Form

☐ Temporary Change	**SOP Name:**	**Digestion of Soils**		**SOP No.:**	**MET10**
☒ Permanent Change	**Analytical Batch/SDG:**	**NA**		**Date:**	**3-8-17**
	Effective Date:	**3-8-17**		**SOP Current Rev.:**	**10**
				Submitted By:	**KMF**
				Approved By:	**JLG**

SOP Section	Change
10.3.21	Change, “To improve Sb, Sn, and/or Ag recovery” to “To improve Sb, Sn, **Ti** and/or Ag recovery.”
10.3.21	Change, “To improve boron recoveries” to “To improve boron and **silicon** recoveries.”
12.0	Add: If a temperature reading is inadvertently not recorded, the digestion batch is still valid based on the historical accuracy of the hot block digestor. Make a note on the digestion log explaining the omission and proceed.

STANDARD OPERATING PROCEDURE FOR HOT BLOCK DIGESTION OF SEDIMENTS AND SOILS

1.0 SCOPE AND APPLICATION

This SOP describes the procedure used by CH2M Hill Applied Sciences Laboratory to digest sediment, soil and other solid samples for analysis by ICP and/or ICPMS. This method follows the guidelines of SW3050B.

2.0 OVERVIEW OF THE ANALYTICAL PROCESS

A homogenized 0.50-1.00 gram aliquot is digested with nitric acid, hydrogen peroxide, and hydrochloric acid as needed. After the digestion process is completed the sample is cooled, diluted to 50 milliliters with ultrapure water, and stored in the polypropylene digestion vessel.
Note the following modifications:

2.1 SW3050B uses 1-2g wet weight sample. This SOP uses 0.50-1.00g aliquots and reduced amounts of reagents.

2.2 SW3050B adds hydrochloric acid based upon which analytical instrument is to be used. This SOP recommends the addition of concentrated HCl based upon which analytes are requested.

3.0 TARGET ANALYTES, REPORTING LIMITS, AND DETECTION LIMITS

3.1 Target analytes: aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, molybdenum, nickel, potassium, selenium, silver, sodium, thallium, vanadium, and zinc.

3.2 All reporting limits are in general at least 2x the laboratory method detection limit. Current method detection limits and reporting limits are available via the Applied Sciences Laboratory MDL database.

3.3 All reporting limits, QC frequency, and QC acceptance criteria are subject to change on a client specific basis as requested by that client.

4.0 INTERFERENCES

Client samples have an extremely wide variety of matrices and each may present unique challenges (i.e. samples high in calcium cause vigorous reactions with the addition of acid, while other samples are difficult to homogenize). Add acids cautiously and slowly if digesting a new matrix to prevent unexpected excessive reactions and loss of sample. Reduced sample size can be used for difficult matrices. Take every precaution to protect samples from contamination from the moment it is poured from the sample container until the time it is covered with the screw cap and sent for analysis.

5.0 SAFETY, WASTE MINIMIZATION, AND POLLUTION PREVENTION

5.1 Laboratory wastes shall be separated and properly disposed of in compliance with all federal, state, and local regulations. These wastes shall be handled according to CVO SOP HAZ01, Waste Disposal.

5.2 Analysts are encouraged to reduce the amount of solvent or disposable labware waste whenever possible. More information on this topic can be found in "Less is Better: Laboratory Chemical Management Waste Reduction" from the American Chemical Society.

5.3 Toxic nitrogen oxide fumes may be evolved; therefore, all work must be performed in a properly operating ventilation system.

5.4 Handle concentrated nitric acid with extreme caution. There is potential for vigorous reactions when the nitric acid is added to the samples. If a vigorous reaction occurs, allow the sample to cool before continuing to the next step. Protective eye wear, gloves, and a protective lab coat must be worn at all times. A face shield and vinyl apron is available as needed.

5.5 Concentrated nitric acid, hydrogen peroxide, and hydrochloric acid must be handled with extreme caution.
5.6 Safety equipment including a fire extinguisher, first aid kit, eye wash, and chemical spill cleanup kit shall be available for use at all times.
5.7 Sample mass and reagent volumes have been reduced to avoid generating excessive amounts of waste.
5.8 Add hydrogen peroxide cautiously and slowly if digesting a new matrix to prevent unexpected excessive effervescing and loss of sample. Hydrogen peroxide is added to completely cooled digestates only.

6.0 SAMPLE COLLECTION, STORAGE, HOLDING TIMES, AND PRESERVATION

6.1 Collect soil samples in clean wide mouth glass containers.
6.2 Store samples at 4°C.
6.3 Holding time is 180 days.
6.4 Digestates are stored at room temperature. Holding time is 180 days.

7.0 APPARATUS AND MATERIALS

All purchasing of apparatus, materials, standards, gases, and reagents is completed according to the ASL SOP31. Support equipment and instrumentation utilized in this SOP and requiring periodic metrological verifications are tracked in ASL's Metrology/Equipment database.

7.1 Analytical balance - capable of weighing to the nearest 0.001g.
7.2 Pipettor – 1-1000uL
7.3 Repipettor – 10 mL adjustable
7.4 HotBlock
7.5 68 mL polypropylene graduated vessel with screw caps
7.6 Disposable ribbed watch glass or reflux cap
7.7 Thermometer – capable of measuring 85-100°C.
7.8 Urethane foam storage racks
7.9 Tongue depressors

8.0 STANDARDS, GASES, AND REAGENTS

8.1 Ultrapure water
8.2 Concentrated nitric acid - trace metals grade
8.3 1+1 nitric acid –Slowly add 60mL concentrated nitric acid to 60 mL UPW
8.4 Concentrated HCl - Optima grade
8.5 Hydrogen Peroxide (30%)
8.6 23 element QC standard. Store at room temperature. Expiration date provided by vendor.
8.7 7 element QC standard. Store at room temperature. Expiration date provided by vendor.
8.8 Ca – 10,000ppm stock. Store at room temperature. Expiration date provided by vendor.
8.9 Mg – 10,000ppm stock. Store at room temperature. Expiration date provided by vendor.
8.10 Na – 10,000ppm stock. Store at room temperature. Expiration date provided by vendor.
8.11 Ca/Mg/Na spike solution. Prepare by pipeting 8.0 mL of each of the single element 10,000ppm stock solutions. Cap and homogenize. Store at room temperature. Expiration date assigned to this solution will be 6 months from the preparation date or the earliest expiration date of the three stock solutions, whichever is earliest.

9.0 QA/QC

9.1 See analytical SOPs MET03 and MET 13 for QAQC acceptance criteria.
9.2 An initial demonstration of capability (IDC) study must be performed prior to use of the method by each analyst or after any significant changes to the method. An IDC study consists of four aliquots of reagent water spiked with target analytes and processed through the entire analytical method.

For NELAC certification purposes the IDC study may be used to satisfy the yearly training requirement for an analyst or work cell.

9.3 A demonstration of capability (DOC) must be performed once per year. Complete a full digestion under observation. The observer should complete the Demonstration of Capability Form found at G:\Controlled Forms\QAQC\DOC Form. This task does not require annual documentation of the preparation and analysis of four spiked samples.

9.4 Work Groups: Analysts are allowed to assist with or perform portions of this SOP without a current IDC or DOC as long as the analyst has read this SOP and is working under the direct supervision of another analyst who has a current IDC or DOC. If an analyst is performing the entire procedure, then the analyst must complete an IDC/DOC.

9.5 Method blank sample is prepared at a rate of 1 set per 20 field samples or one per batch, whichever is greater.

9.6 Laboratory control sample (LCS) is prepared at a rate of 1 set per 20 field samples or one per batch, whichever is greater. Solutions used to spike the LCS are also used to prepare calibration standards.

9.7 Matrix spike/spike duplicate samples, (MS/MSD) are prepared at a rate of 1 set per 20 field samples or one per batch, whichever is greater. MS/MSD samples should be chosen randomly from a client batch of samples unless they are pre-selected by the client. Analysts should rotate the client selected for matrix spikes so that recovery and precision data is collected from a wide variety of sample matrices. Note that MS/MSD samples are not prepared for ICPMS analysis. Typically soil samples require dilution before running on the ICPMS, so the higher MS/MSD spike concentrations found in the MS/MSD samples prepared for ICP analysis are appropriate

9.8 Digestates are stored in the same vessels they are digested in to reduce the risk of contamination.

9.9 Uncertainty of measurements: Calculated by following ASL SOP30.

9.10 The major sources of error in this method are as follows: homogenization of sample, weighing samples and introduction of contamination during digestion.

9.11 Limit of Detection/Limit of Quantitation: See ASL SOP 32.

9.12 Markings on digestion vessels are verified for accuracy prior to use for DoD samples. For each lot of vessels, document the accuracy of the vessel markings using the form located at: G:\\Metals\DigestionVesselValidation.

10.0 PROCEDURE

10.1 Digestion Vessels

Vessels should be ready to use as received from the vendor; however, if contamination is found and/or suspected the vessels should be pre-cleaned. This can be done by pouring 5% nitric solution in the vessels and heating it in the hot block for several hours. This may be necessary if zinc by ICPMS is requested with a reporting limit at or below 5ppb.

10.2 Bench Sheet

Document digestions on bench sheets located at G:\\Controlled Forms\Metals\. Complete the header information appropriately. Label the digestion batch as the date plus a letter (i.e. 050810A = first batch of the day). Record the lot numbers of the vessels, acids and spike solutions. Record the sample ID's as follows:

SB1-0508	(SB2-0508 if this is the second digestion batch of the day)
BS1S0508	(BS2S0508 if this is the second digestion batch of the day)
STG3475-01	(ST = Soil Total)
STG3475-01MS	(Matrix Spike)
STG3475-01MSD	(Matrix Spike Duplicate)

Record the time and temperature when placing samples on and off the hot block.

10.3 Digestion procedure

10.3.1 Calibrate the balance using certified weights and record in logbook. The weights must bracket the working range. See ASL SOP15.

10.3.2 Preheat the HotBlock ~1 hour to stabilize the temperature at 95ºC $\pm$5ºC.

10.3.3 Mix the sample thoroughly using a tongue depressor to achieve homogeneity.

10.3.4 Place a digestion vessel on the balance and tare.

10.3.5 Sub-sampling prior to analysis will be completed following the procedures in the ASL SOP 40. Weigh (to the nearest 0.001g) 0.500-1.000g aliquot directly into vessel. Increase sample mass if high in moisture.

10.3.6 Add 2 glass beads to SB and BS.

10.3.7 Spikes for ICP analysis: Add spikes to vessels labeled LCS, MS and MSD as follows: 250uL of the 23 element and 7 element QC standards, 143ul of the Ca/Mg/Na spike solution.

10.3.8 Spikes for ICPMS analysis: Add spikes to vessel labeled LCS as follows: 250uL of 1:10 dilutions of the 23 element and 7 element QC standards. A separate ICPMS MS/MSD is not required. A dilution of the ICP MS/MSD digestate is made at the instrument.

10.3.9 Working in the hood, dispense ~2.5mL UPW and 2.5mL concentrated HNO_3 into the vessels. Swirl and cover with reflux caps.

10.3.10 Reflux for at least 15 minutes. Cool.

10.3.11 Dispense 2.5 mL concentrated HNO_3.

10.3.12 Reflux for at least 30 minutes. Place thermometer in a vessel which closely mimics sample conditions, as deemed appropriate. The digestion start time should be recorded when the thermometer reads 95ºC $\pm$5ºC. Cool.

10.3.13 If brown fumes were generated during the previous step, repeat 10.3.9 (omitting the water addition) and 10.3.10 until no brown fumes are emitted to ensure complete oxidation.

10.3.14 Reflux at least 1 1/2 hours. The vessel must not go dry (re-digest sample if the vessel goes dry). Place thermometer in a vessel which closely mimics sample conditions, as deemed appropriate. The digestion start time should be recorded when the thermometer reads 95ºC $\pm$5ºC.

10.3.15 Cool completely.

10.3.16 Add ~2-5mL UPW and 0.5mL 30% hydrogen peroxide slowly (vigorous reaction possible).

10.3.17 Heat until the effervescence subsides. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Lift vessel out of the hot block if necessary. Cool.

10.3.18 Continue to add 30% hydrogen peroxide in 0.5 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (Note: a total of 1.5 mL is normal, do not exceed 5mL 30% H_2O_2).

10.3.19 Reflux with hydrogen peroxide for at least 30 minutes. Place thermometer in a vessel which closely mimics sample conditions, as deemed appropriate. The digestion start time should be recorded when the thermometer reads 95ºC $\pm$5ºC. Cool.

10.3.20 Bring the final volume to 50mL using the markings on the digestion vessels with UPW. The acids increase the density of the digestates. Screw on green caps and homogenize. Allow digestates to settle overnight prior to analysis.

10.3.20.1 If the digestion vessel is overfilled past the 50 mL mark, carefully pour the digestate into a clean Class A graduated cylinder and record the volume. Transfer the digestate back to the digestion vessel. Rinse the graduated cylinder three times with UPW.

10.3.21 To improve Sb, Sn, and/or Ag recovery, the addition of concentrated HCl is recommended in SW3050 section 7.5 as follows: After step 10.3.8, add 5.0mL concentrated HCl and 1.25mL concentrated HNO_3. Place thermometer in a vessel which closely mimics sample conditions, as deemed appropriate. The digestion start time should be recorded when the thermometer reads 95ºC $\pm$5ºC. Reflux for at least 1 hour. Cool and follow step 10.3.20 to finish. Digesting soils this way for antimony will improve MS/MSD recoveries from~10% to ~90%. Insoluble oxides form when soils are digested with only nitric acid. To improve boron recoveries, an additional 20mL UPW may be added to the digestion vessel in step 10.3.9.

11.0 DATA REDUCTION

Transcriptions of hand generated data (soil sample masses) are reviewed by the analyst when entered into the electronic reporting system.

12.0 DOCUMENTATION

Completed digestion bench sheets are stored in the Digestion Notebook in the Metals Laboratory. Digestion bench sheets will be initialed and dated by a peer after a review has been completed. After a minimum of 1 year the digestion bench sheets are moved to an offsite record center, see ASL SOP17 Data Storage.

13.0 REFERENCES

13.1 Test Methods for Evaluating Solid Waste, EPA SW-846 Method 3050B.
13.2 "EPA Method 3050B for use with the HotBlock" prepared by Environmental Express.

14.0 DEFINITIONS

14.1 ASL – Applied Sciences Laboratory
14.2 ASLRT – Applied Sciences Laboratory Reporting Tool
14.3 CVO – Corvallis, OR
14.4 NELAC – National Environmental Laboratory Accreditation Conference
14.5 NELAP – National Environmental Laboratory Accreditation Program
14.6 QA/QC – Quality Assurance/Quality Control
14.7 QA – Quality Assurance
14.8 QC – Quality Control
14.9 SOP – Standard Operating Procedure
14.10 IDC – Initial Demonstration of Capability
14.11 RSD – Relative Standard Deviation
14.12 %D – Percent Difference
14.13 LCS – Laboratory Control Standard
14.14 LCSD – Laboratory Control Standard Duplicate
14.15 Laboratory Duplicates (Dup) – Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of duplicates indicate precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.
14.16 Field Duplicates (FD) – Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedure. Analyses of Duplicates gives a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.
14.17 Laboratory Reagent Blank (WB1, SB1, XB1) – An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The blank is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
14.18 Blank Spike (BS1W, BS1S) – An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.
14.19 Matrix Spikes/Spike Duplicates (MS/MSD) – An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS/MSD is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.
14.20 Stock Standard Solution (SSS) – A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.
14.21 Primary Standard Solution (PSS) – A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.

CHANGE HISTORY

1. Changes made in revision 4

1.1 Under APPARATUS AND MATERIALS capability of the analytical balance has been changed from 0.01g to 0.001g.
1.2 In section 10.3.6 the addition of spikes has been included so that the samples are spiked prior to the addition of acid.
1.3 In section 10.4 and section 10.5 glass beads are used in the LCS and method blank vessels. Previously empty vessels were spiked.
1.4 In Exhibit A the ICP spikes have been changed. Instead of 500uL of 23 element QC standard and the 7 element QC standard, 250uL of each is used. Instead of 135uL of 3333ppm Ca/.Mg/Na spike solution, 143uL is now used. Final spike levels for the majority of elements are at 500ppb rather than 1000ppb.
1.5 A new benchsheet has replaced the old one in Exhibit B.

2. Changes made in revision 5

2.1 Section 2.0: Digestate dilution comment removed. Applicable to SOPs MET 03 and MET 13.
2.2 Section 6.0: Added holding time of digestates.
2.3 Section 8.0: Removed 1 + 4 nitric because it is not used.
2.4 Section 8.0: Added storage and expiration of spike solutions
2.5 Section 9.0: Added reference to the analytical SOPs, IDC acceptance criteria, DOC requirement.
2.6 Section 9.7: Reagent and Equipment Quality Control/Contamination Logs implemented.
2.7 Section 10.1: Explained the use of pre-cleaned vessels.
2.8 Section 10.2: Reagent and Equipment Quality Control/Contamination Logs implemented.
2.9 Section 10.3.18 changed the reference of 10.3.15 to 10.3.6.
2.10 Section 10.4, 10.5 and 10.6 were combined with 10.3.6.
2.11 Section 12.0: Added information about offsite storage of digestion benchsheets.
2.12 Section 12: Reagent and Equipment Quality Control/Contamination Logs implemented.
2.13 Exhibit B: Updated in response to the Arizona audit. Final volume/weight column altered.

3. Changes made in revision 6

3.1 The approval signature block was updated from the QA officer and the author to the author and the section leader.
3.2 Section 7.0: Add reference to the Purchasing SOP and Metrology/Equipment matrix.
3.3 Sections 9.8 and 9.9: Separation of "Source of error" and "uncertainty SOP reference".
3.4 Section 10.2: Add reference to controlled form.
3.5 Section 10.3.5: Add subsampling SOP reference per DoD audit
3.6 Section 10.3.9: Changed volume of nitric acid from 5mL to 2.5mL.
3.7 Section 10.3.16: Increased digestion time from 30 minutes to 2 hours.
3.8 Section 10.3.17: Bring final volume to 50mL using the balance instead of the vessel gradations.
3.9 Section 11.0: Data integrity addressed.
3.10 Exhibit B: Removed digestion benchsheet.

4. Changes made in revision 7

4.1 Updated the signature block on the cover page.
4.2 Section 7.0: Updated reference to the Metrology/Equipment database
4.3 Sections 8.1: removed reference to Millipore water
4.4 Keep section 3.2 reference to MDLs since the studies are required for certification in some states
4.5 Sections 7.0 and 8.0: removed vendor names and part numbers
4.6 Removed exhibit A and incorporated into body of section 10.3
4.7 Section 10.3.15: change to " add 2-5 mL UPW and 0.5mL 30% H2O2 slowly"
4.8 Section 10.3.18: change from 2 hours to 30 minutes as per Environmental Express method.
4.9 Section 10.3.20: added note on how to improve boron recoveries

5. Changes made in revision 8

5.1 Updated the signature block on the cover page.
5.2 Section 2.1: Included reduced reagent volumes.
5.3 Added Section 9.4 describing work groups.
5.4 Section 9.12: Updated the procedure for documenting the cleanliness of newly purchased acids and reaction vessels.
5.5 Section 10.3.9: Change to "dispense ~2.5mL UPW and 2.5mL concentrated HNO_3 into the vessels." Corrected grammar.
5.6 Section 10.3.11: Change to "dispense 2.5mL concentrated HNO_3."
5.7 Section 10.3.20: Change to "the addition of concentrated HCl is recommended in SW3050 section 7.5 as follows: After step 10.3.8, add 5.0mL concentrated HCl and 1.25mL concentrated HNO_3."
5.8 Section 2.2: Change to "the addition of concentrated HCl."
5.9 Section 10.3.10, 12, 14, 19 and 21: Updated the procedure describing when to begin timing digestion.

6. Changes made in revision 9

6.1 Updated the signature block on the cover page.
6.2 Added Section 10.3.20.1.
6.3 In Section 10.3.20, changed, "Bring the final volume to 50mL by placing the vessel on the tared balance and bringing the mass to 51.0 ± 0.5g using UPW" to read "Bring the final volume to 50mL using the markings on the digestion vessels with UPW."
6.4 Added Section 9.13

7. Changes made in revision 10

7.1 Updated the signature block on the cover page.
7.2 Section 10.3.21: Changed "Cool and follow step 10.3.19" to "Cool and follow step 10.3.20."
7.3 Section 10.3.10: Removed reference to recording digestion time. Insert, "Reflux at least 15 minutes."
7.4 Sections 10.3.12, 10.3.14, 10.3.19, 10.3.21: Inserted the phrase "at least" in sentences describing the length of time to reflux samples.
7.5 Section 9.7: Inserted note explaining that ICPMS MS/MSD samples are not prepared.
7.6 Deleted section 9.12. Referred to tracking reagent lots in a log book.
7.7 Section 10.3.8: Rewrote section. Removed reference to preparation of ICPMS MS/MSD samples.
7.8 Section 9.3: Changed DOC requirements.
7.9 Section 10.2: Removed reference to location of time/temperature fields on digestion bench sheet. Removed reference to reagent log book, and verifying vessel cleanliness.
7.10 Section 10.3.13: Inserted "omitting the water addition."
7.11 Section 12.0: Removed reference to tracking reagent lots in a log book.